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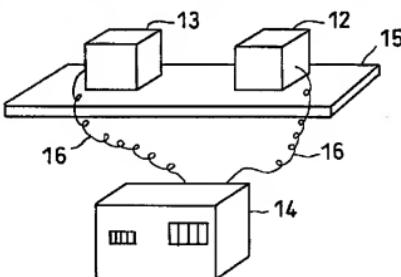
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(54)【発明の名称】 金属の酸化物又は水酸化物のパイプ内壁付着量測定装置、付着状況評価方法、及び付着状況予測方法

(57)【要約】

【課題】 パイプの内壁に付着した金属の酸化物又は水酸化物の付着量を測定するパイプ内壁付着量測定装置、付着状況を評価する評価方法、及び付着状況を予測する予測方法を提供する。

【解決手段】 照射部12と受光部13との間に被測定物であるテフロン(登録商標)パイプテフロンパイプ(図示せず)を置き、テフロンパイプの外側から光を当て、透過した光を受光部13で受光して電圧に変換し、制御・表示部14で透過光量を表示する。この透過光量によりテフロンパイプの内壁に付着した金属酸化物の付着量を測定することができる。また、経年的に測定することにより、将来の金属酸化物の付着状況を予測することもできる。



## 【特許請求の範囲】

【請求項1】サイリスタバルブの冷却水の配管に使用される透明または半透明のパイプの内壁に付着する金属の酸化物又は水酸化物の付着量を、パイプの外側から光を当て、その光の透過光量により測定することを特徴とする金属の酸化物又は水酸化物のパイプ内壁付着量測定装置。

【請求項2】サイリスタバルブの冷却水の配管に使用される透明または半透明のパイプの外側から光を照射する照射手段と、この手段から照射され、前記パイプを透過してきた光を受光して電圧に変換する受光手段と、前記照射手段から照射される光の強さを設定すると共に前記受光手段により受光した光の量を表示する手段とを備えたことを特徴とする金属の酸化物又は水酸化物のパイプ内壁付着量測定装置。

【請求項3】前記照射手段からパイプに照射する光の光軸が前記受光手段の受光面の中心に垂直になるように保ち、かつ、照射し受光する光路の距離を一定に保った状態となるように前記照射手段及び前記受光手段を配置したことを特徴とする請求項2に記載の金属の酸化物又は水酸化物のパイプ内壁付着量測定装置。

【請求項4】前記照射手段の光を照射する端部、及び、前記受光手段の光を受光する端部の少なくとも一方を、前記パイプの外壁に接するように配置したことを特徴とする請求項3に記載の金属の酸化物又は水酸化物のパイプ内壁付着量測定装置。

【請求項5】前記照射手段は光源としてレーザを用い、前記受光手段はレーザ光を受光して電圧に変換する素子を有することを特徴とする請求項4に記載の金属の酸化物又は水酸化物のパイプ内壁付着量測定装置。

【請求項6】前記照射手段は光源としてハロゲンランプを用い、前記受光手段はハロゲンランプの光を受光して電圧に変換する素子を有することを特徴とする請求項4に記載の金属の酸化物又は水酸化物のパイプ内壁付着量測定装置。

【請求項7】前記ハロゲンランプの光を受光して電圧に変換する素子に前記ハロゲンランプからの光だけが入るように、他の光を遮蔽する遮蔽手段を備えたことを特徴とする請求項6に記載の金属の酸化物又は水酸化物のパイプ内壁付着量測定装置。

【請求項8】前記照射手段の光を照射する端部、及び、前記受光手段の光を受光する端部を前記パイプの外周に沿って移動させる手段を備えたことを特徴とする請求項4に記載の金属の酸化物又は水酸化物のパイプ内壁付着量測定装置。

【請求項9】前記照射手段の光を照射する端部、及び、前記受光手段の光を受光する端部を前記パイプの長さ方向に沿って移動させる手段を備えたことを特徴とする請求項4に記載の金属の酸化物又は水酸化物のパイプ内壁

【請求項10】パイプに金属の酸化物又は水酸化物が付着している長さと、金属の酸化物又は水酸化物が付着した部分の透過光量の値とから、金属の酸化物又は水酸化物のパイプへの付着状況を評価することを特徴とする金属の酸化物又は水酸化物のパイプ内壁付着状況評価方法。

【請求項11】パイプに金属の酸化物又は水酸化物が付着している長さと、金属の酸化物又は水酸化物が付着した部分の透過光量の値とを定期的に測定し、その時間的な傾向から金属の酸化物又は水酸化物のパイプへの付着状況を予測することを特徴とする金属の酸化物又は水酸化物のパイプ内壁付着状況予測方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、水冷サイリスタバルブの冷却水配管に使用されるパイプの内壁に付着する金属の酸化物又は水酸化物の付着量を測定するパイプ内壁付着量測定装置、付着状況を評価する評価方法、及び付着状況を予測する予測方法に関する。

【0002】

【従来の技術】水冷サイリスタバルブは、サイリスタ素子やアノードリアクトルという発熱する部品を絶縁抵抗値の高い水、いわゆる純水で冷やしている。このような発熱部品の金属部分が直接、冷却水と接している部分では、金属部分の金属が冷却水中に僅かではあるが溶出し、冷却水中の溶存酸素と反応して金属の酸化物あるいは水酸化物となり、冷却水中に存在している。

【0003】以下、図7及び図8を参照して、水冷サイリスタバルブにおける冷却水の配管経路を説明する。図7は水冷サイリスタバルブにおける配管構造を示す図であり、図8は図7で示した各モジュールにおける配管構造を示す図である。

【0004】図7において、サイリスタバルブは、モジュール1を四段に積み重ねた構造になっており、各モジュール1（1a～1h）間は少なくとも1本の絶縁配水管2で繋がっている。冷却水として、サージタンク3に貯まっている水をポンプ4で配水管5と絶縁配水管2を経由してモジュール1aに送り、更に絶縁配水管2を経由してモジュール1bからモジュール1hまで順次、巡っていく。冷却水の温度は熱交換器4で、電導度はイオン交換器5で、それぞれ所定の温度と電導度になるようにコントロールされる。また、配水管5の要所にはストレーナ8を設けて冷却水中の固形物を捕捉している。

【0005】モジュール1においては、図8に示したように、サイリスタSの冷却体9、アノードリアクトルJの冷却体10、抵抗器Rの冷却体11が、水冷用品として使われている。冷却水は、モジュール1内では配水管25の入口INから入ってこれらの水冷用品を巡った後、配水管25の出口OUTを経由して出していく。

絶縁性の高い水、すなわち電気電導度の非常に低い水（水の電導度が  $1 \mu S/cm$  以下）が用いられる。冷却水の中には、配水管やこれらの水冷用品の金属部材から溶けた金属イオンとその酸化物である微粒子が含まれる。特に、サイリスタ S の冷却体 9 とアノードリニアクトルルの冷却体 10 は熱伝導率や電気伝導率の点から銅が使用されるため、冷却水中の金属酸化物として銅の酸化物が多く含まれることになる。

#### 【0007】

【発明が解決しようとする課題】ところで、上述のようなサイリスタバルブにおいては、これら金属イオンとその酸化物はイオン交換器 7 に詰めてあるイオン交換樹脂やストレーナ 8 等にトラップされるが、長期間稼動している間にはモジュール 1 間の絶縁配水管 2 やモジュール 1 内の絶縁配水管 2 の内壁に付着してくる。

【0008】絶縁配水管 2、2 は、冷却水への影響を極力少なくするため、材料としてテフロンを用いたテフロンパイプが用いられる。銅の金属酸化物がテフロンパイプに付着した場合、その付着した状態によってはテフロンパイプの絶縁特性が懸念される。

【0009】テフロンパイプは半透明であることから、テフロンパイプの内壁に付着した銅の金属酸化物は外観から黒く見えるため、従来、その黒く見える度合いを肉眼で判断して金属酸化物の付着程度を評価していた。

【0010】しかし、肉眼で判断して評価する事は個人差があるため、正確さや適正さに欠ける。

【0011】そこで、本発明は、従来のこのようない点に鑑みがされたもので、パイプの内壁に付着した金属の酸化物又は水酸化物の付着量を測定するパイプ内壁付着量測定装置、付着状況を評価する評価方法、及び付着状況を予測する予測方法を提供することを目的とする。

#### 【0012】

【課題を解決するための手段】上記目的を達成するため、請求項 1 に記載の本発明に係る金属の酸化物又は水酸化物のパイプ内壁付着量測定装置は、サイリスタバルブの冷却水の配管に使用される透明または半透明のパイプの内壁に付着する金属の酸化物又は水酸化物の付着量を、パイプの外側から光を当て、その光の透過光量により測定することを特徴とする。

【0013】請求項 1 に記載の本発明によれば、パイプの内壁に付着した金属の酸化物又は水酸化物の付着を、パイプの外側から光を当てて、その光の透過光量の数値で評価するため、パイプの内壁に付着する金属酸化物の付着量を適正に測定し、評価することができる。

【0014】請求項 2 に記載の本発明に係る金属の酸化物又は水酸化物のパイプ内壁付着量測定装置は、サイリスタバルブの冷却水の配管に使用される透明または半透明のパイプの外側から光を照射する照射手段と、この手段から照射され、パイプを透過してきた光を受光して電

強さを設定すると共に受光手段により受光した光の量を表示する手段とを備えたことを特徴とする。

【0015】請求項 2 に記載の本発明によれば、予め強さを調節した光により、パイプを透過した光の量を電圧に変換することができるので、この電圧値により受光量を数値で表すことができる。

【0016】請求項 3 に記載の本発明は、請求項 2 に記載の金属の酸化物又は水酸化物のパイプ内壁付着量測定装置において、照射手段からパイプに照射する光の光軸が前記受光手段の受光面の中心に垂直になるように保持し、かつ照射し受光する光路の距離を一定に保った状態となるように照射手段及び受光手段を配置したことを特徴とする。

【0017】請求項 3 に記載の本発明によれば、照射する光の光軸が受光面の中心に垂直になるように保持し、かつ、照射し受光する光路の距離を一定に保った状態でパイプの内壁に付着する金属の酸化物又は水酸化物の付着をパイプの外側から光を当ててその光の透過光量で測定することになる。これは、光量（光のエネルギー）は照射した光の光軸が受光面上の角度とその光路の距離に依存するためである。照射する光の光軸が受光面の中心に垂直に照射される時に受光面での光量は最大になるからである。また、光路の距離を一定に保つのは、光路の距離が変わると受光面での透過光量が変わるために、透過光量がパイプの内壁に付着した金属の酸化物又は水酸化物の量に従ってのみ変化するようにするためである。

【0018】請求項 4 に記載の本発明は、請求項 3 に記載の金属の酸化物又は水酸化物のパイプ内壁付着量測定装置において、照射手段の光を照射する端部、及び、受光手段の光を受光する端部の少なくとも一方を、パイプの外壁に接するように配置したことを特徴とする。

【0019】請求項 4 に記載の本発明によれば、光を照射する端部、及び、光を受光する端部の少なくともいずれかをパイプの外壁に接するようにした状態でパイプの透過光量を測定することにより、パイプを透過させる光の量の、光路の距離による減衰を最少にすることができます。

【0020】請求項 5 に記載の本発明は、請求項 4 に記載の金属の酸化物又は水酸化物のパイプ内壁付着量測定装置において、照射手段は光源としてレーザを用い、受光手段はレーザ光を受光して電圧に変換する素子を有することを特徴とする。

【0021】請求項 5 に記載の本発明において、光源としてレーザを用いるのはレーザというエネルギーの大きな光によって半透明なパイプでも透過光量を大きくして金属の酸化物又は水酸化物が付着した時の付着量の違いが透過光量の変化に大きく現れるようにするためである。さらに、受光した光を電圧に変換する素子に光源のレーザに反応するものを用いるため、受光した透過光量

受けたことがない。

【0022】請求項6に記載の本発明は、請求項4に記載の金属の酸化物又は水酸化物のパイプ内壁付着量測定装置において、照射手段は光源としてハロゲンランプを用い、受光手段はハロゲンランプの光を受光して電圧に変換する素子を有することを特徴とする。

【0023】請求項6に記載の本発明によれば、ハロゲンランプを光源とし、その光を受光して電圧に変換する素子を用いるため、比較的安価で強力な光量による測定を行うことができる。

【0024】請求項7に記載の本発明は、請求項6に記載の金属の酸化物又は水酸化物のパイプ内壁付着量測定装置において、ハロゲンランプの光を受光して電圧に変換する素子にハロゲンランプからの光だけが入るように、他の光を遮蔽する遮蔽手段を備えたことを特徴とする。

【0025】請求項7に記載の本発明によれば、ハロゲンランプ以外からの光がパイプに入射して受光する部分に入らないように、外光を遮蔽する遮蔽手段を設けたことにより、ハロゲンランプ以外の光の影響を抑制することができる。

【0026】請求項8に記載の本発明は、請求項4に記載の金属の酸化物又は水酸化物のパイプ内壁付着量測定装置において、照射手段の光を照射する端部、及び、受光手段の光を受光する端部をパイプの外周に沿って移動させる手段を備えたことを特徴とする。

【0027】請求項8に記載の本発明によれば、光を照射する端部、及び、光を受光する端部パイプの外周に沿って移動させて透過光量を測定することにより、パイプの外周に沿った各位置における金属の酸化物又は水酸化物の付着量を測定することができる。

【0028】請求項9に記載の本発明は、請求項4に記載の金属の酸化物又は水酸化物のパイプ内壁付着量測定装置において、照射手段の光を照射する端部、及び、受光手段の光を受光する端部をパイプの長さ方向に沿って移動させる手段を備えたことを特徴とする。

【0029】請求項9に記載の本発明によれば、光を照射する端部、及び、光を受光する端部をパイプの長さ方向に沿って移動させて透過光量を測定することにより、テフロンパイプの長さ方向の各位置における金属の酸化物又は水酸化物の付着量、及び付着している長さを測定することができる。

【0030】請求項10に記載の本発明に係る金属の酸化物又は水酸化物のパイプ内壁付着状況評価方法は、パイプに金属の酸化物又は水酸化物が付着している長さと、金属の酸化物又は水酸化物が付着した部分の透過光量の値とから、金属の酸化物又は水酸化物のパイプへの付着状況を評価することを特徴とする。

【0031】請求項10に記載の本発明によれば、金属

備することができる。

【0032】請求項11に記載の本発明に係る金属の酸化物又は水酸化物のパイプ内壁付着状況予測方法は、パイプに金属の酸化物又は水酸化物が付着している長さと、金属の酸化物又は水酸化物が付着した部分の透過光量の値とを定期的に測定し、その時間的な傾向から金属の酸化物又は水酸化物のパイプへの付着状況を予測することを特徴とする。

【0033】請求項11に記載の本発明によれば、金属の酸化物又は水酸化物のパイプへの今後の付着状況を適正に予測することができる。

【0034】

【発明の実施の形態】以下、図面を参照して本発明の実施形態について詳細に説明する。

【0035】図1は、本発明の一実施形態に係る金属の酸化物又は水酸化物のパイプ内壁付着量測定装置の概略構成を示す図である。

【0036】この装置は主として三つの部分から構成され、12は光を照射する照射部、13は照射部12に対向して配置され受光した光を電圧に変換する受光部、14は照射する光の強さを設定すると共に受光した光の量を表示する制御・表示部である。

【0037】照射部12から照射した光の光軸が受光部13の受光面の中心に垂直になるように、図1では光源部12と受光部13を固定する固定板15を使用し、照射し受光する光路の距離を一定に保った状態となるようしている。このように照射する光の光軸が受光面の中心に垂直になるようにすることにより、受光面での光量は最大になる。また光路の距離を一定に保つのは、光路の距離が変わると受光面での透過光量が変わるために、透過光量がテフロンパイプの内壁に付着した金属酸化物の量に従ってのみ変化するようにするためである。

【0038】測定されるテフロンパイプ（例えば図7のテフロンパイプ2、または図8のテフロンパイプ22、図1では図示せず）は照射部12と受光部13の間に置かれる。テフロンパイプの内壁に付着した金属酸化物はテフロンパイプを透過する光の透過を妨げるため、テフロンパイプの内壁に付着した金属酸化物の量にテフロンパイプを透過する光の量は依存する。なお、照射部12の光を照射する端部、または受光部13の光を受光する端部を、テフロンパイプの外壁に接するように配置して測定することにより、テフロンパイプを透過させる光の量の、光路の距離による減衰を最少にすることができる。

【0039】照射部12から照射された光がテフロンパイプを透過して受光部13で受光して電圧に変換される。電圧に変換された受光量は、制御・表示部14においてボルト単位で表示したり、また、初期の測定値（例えば、金属酸化物の付着していないテフロンパイプで測

パイプの内壁に金属酸化物が付着した場合の測定値をこの初期値に対する百分率（パーセント）で表示したりすることが可能になる。

【0040】図2は、テフロンパイプ2'（例えば図7のテフロンパイプ2'または図8のテフロンパイプ2'に対応する。）の内壁に金属酸化物が付着した時の測定方法の例を説明するためのものである。ここでは照射部1'2と受光部1'3と固定板1'5のみを図示した。

【0041】金属酸化物が付着したテフロンパイプを測定する方法としては、図2に示すように照射部1'2と受光部1'3が固定板1'5で一体となったものを、単に、金属酸化物が付着している箇所において測定する第1の方法、照射部1'2と受光部1'3が固定板1'5で一体となったものを矢印aで示したようにテフロンパイプ2'の外周方向にずらしながらテフロンパイプ2'の全周にわたって測定する第2の方法、また、照射部1'2と受光部1'3が固定板1'5で一体となったものを矢印bで示したようにテフロンパイプ2'の長さ方向にずらしながら測定する第3の方法がある。

【0042】第2の方法によれば、テフロンパイプ2'の外周に沿った各位置における金属酸化物の付着量を測定することができる。

テフロンパイプの種類	透過光量測定値（%）
テフロンパイプ（酸化銅付着無し）	6.54～7.42
テフロンパイプ（酸化銅付着有り）	0.27～0.42

【0046】また、経年に第1～第3の方法による測定を実施することにより、その測定値の時間的な傾向から将来の金属酸化物の付着状況を予測したテフロンパイプ2'の特性評価が可能になる。

【0047】例えば第3の方法により、テフロンパイプ2'の長さ方向の各位置における金属酸化物の付着量（透過光量）を経年的に測定すれば、テフロンパイプ2'の長さ方向の各位置における将来の金属酸化物の付着量（透過光量）が図3に示すように測定されたとすると、例えば、5年後および10年後、15年後の長さ方向の各位置における金属酸化物の付着量（透過光量）が図4のようにプロットすることにより、20年後、25年後に付着している長さL<sub>20</sub>、L<sub>25</sub>を予測することができる。

【0048】以上のように、本実施形態によれば、内壁に金属酸化物が付着したテフロンパイプ2'における金属酸化物の付着状態を評価するのに、テフロンパイプ2'の外側から光を照射し、その光の透過光量を測定して評価するため、今まで肉眼で判断して評価していたことに比べると、個人差が無くなり、正確さと適正さが大幅に向かう。

【0049】また、測定結果を数値で表すことが可能と

【0043】また、第3の方法によれば、テフロンパイプ2'の長さ方向（長手方向）の各位置における金属酸化物の付着量、及び付着している長さを測定することができ、これらの測定結果によりテフロンパイプ2'への金属酸化物の付着状況を評価することにより、テフロンパイプ2'の特性評価が可能になる。

【0044】表1は、テフロンパイプ2'の内壁に酸化銅の粉体が付着した物と付着していないものを、第2の方法で測定した透過光量測定例を示したものである。ただし、テフロンパイプ2'を除いた時の透過光量測定値を100.00（%）とした。ここでは照射部1'2の光源としてレーザ光を発光する光源を、受光部1'3にはこのレーザの波長にのみ感知する素子を用いた。光源としてレーザを用いることによりレーザというエネルギーの大きな光によって半透明なテフロンパイプでも透過光量を大きくして金属酸化物が付着した時の付着量の違いが透過光量の変化に大きく現れるようにすることができる。表1に示すように、テフロンパイプ2'の内壁に酸化銅の粉体が付着することにより、透過光量が減少することがわかる。

【0045】

【表1】

とにより、将来の金属酸化物の付着状況を予測したテフロンパイプ2'の特性評価が可能になる。

【0050】なお、テフロンパイプ2'に照射する光やテフロンパイプ2'を透過してきた光を受光する際の光路を自在に取り扱えるように光ファイバーを用いてもよい。この場合、照射部1'2に第1の光ファイバーの一端を接続すると共にその他端をテフロンパイプ2'の外側の被測定箇所に接続させ、テフロンパイプ2'におけるこの箇所と対向する箇所に第2の光ファイバーの一端を接続させ、その他端を受光部1'3に接続すればよい。

【0051】また、ここまでは、半透明のテフロンパイプを対象にして述べたが、他の半透明のパイプ、例えばポリエチレンパイプや、透明のパイプでも本実施形態の適用は可能である。

【0052】次に、図5及び図6を用いて、本発明の他の実施形態に係る金属酸化物パイプ内壁付着量測定装置について説明する。

【0053】この実施形態は、照射部1'2の光源として、比較的安価で強力な光量による測定を行うため例えばハロゲンランプを使用した場合、測定の邪魔になる光を遮蔽するため、測定するテフロンパイプ2'の部分、照射部1'2、及び、受光部1'3を覆う遮蔽物1'7を設けたものであり、図5はその主要部の外観を示す正面図、

おいて c の部分は遮蔽物 17 がテフロンパイプ 2' を被った時の合わせ目であり、d の箇所がヒンジになっている。

【0054】本実施形態によれば、ハロゲンランプ以外からの光がパイプに入射して受光する部分に入らないように、外光を遮蔽する遮蔽物を設けたことにより、ハロゲンランプ以外の光の影響を抑制することができる。

[0055]

【発明の効果】以上のように、本発明によれば、内壁に金属の酸化物又は水酸化物が付着したパイプにおける金属の酸化物又は水酸化物の付着状態を評価するのに、パイプの外側から光を照射し、その光の透過光量を測定して評価するため、今まで肉眼で判断して評価していたことに比べると、個人差が無くなり、正確さと適正さが大幅に向こむ。

【0056】また、測定結果を数値で表すことが可能となるため、経年的に金属の酸化物又は水酸化物の付着状況を測定することにより、将来の金属の酸化物又は水酸化物の付着状況を予測したテフロンパイプの特性評価が可能になる。

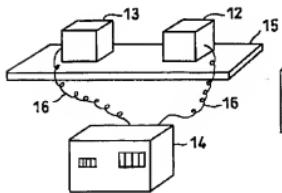
### 【図面の簡単な説明】

【図1】 本発明の一実施形態に係る金属の酸化物又は水酸化物のパイプ内壁付着量測定装置の概略構成を示す斜視図。

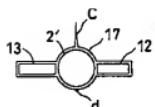
【図2】 一実施形態における金属酸化物の付着したテフロンパイプの測定方法の例を説明するための斜視図。

【図3】 一実施形態におけるテフロンパイプの長さ方向の各位置における金属酸化物の付着量の経年変化を示す図。

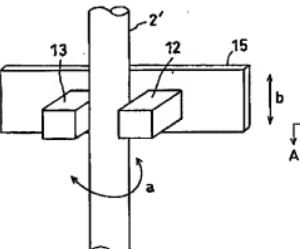
【図1】



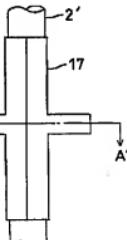
〔图6〕



## 【図2】



【図5】



【図4】 一実施形態における将来の金属酸化物の付着している長さを予測することを説明するための図。

【図5】 本発明の他の実施形態に係る金属の酸化物又は水酸化物のパイプ内壁付着量測定装置の主要部の外観を示す正面図

【図6】図5のA-A'断面図-

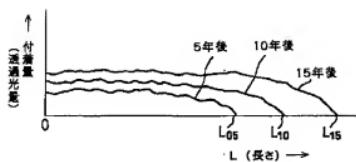
【図7】 水冷サイリスタバルブにおける配管構造を示す図

【図8】 図7で示した水冷サイリスタバルブの各モジュールにおける配管構造を示す図

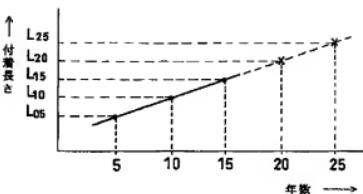
### 【符号の説明】

- 1 …モジュール
- 2'、2、22…絶縁配管
- 3…サーボタンク
- 4…ポンプ
- 5、2 5…配水管
- 6…熱交換器
- 7…イオン交換器
- 8…ストレーナ
- 9…サイリスタの冷却体
- 1 0…アノードリアクトルの冷却体
- 1 1…抵抗器の冷却体
- 1 2…照射部
- 1 3…受光部
- 1 4…制御・表示部
- 1 5…固定板
- 1 6…ケーブル
- 1 7…遮蔽物

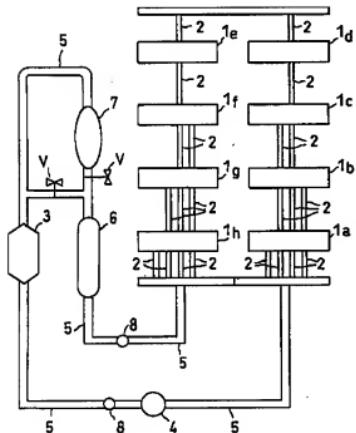
【图3】



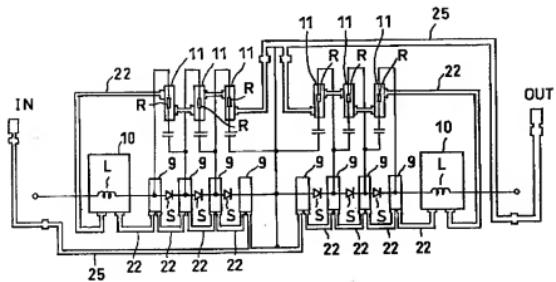
【图 4】



【图7】



【図8】



フロントページの続き

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FF02 GG02 GG04 HH15 LL02  
MM04 MM07 PP01 SS11  
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CD03 CD06 FA10

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C07C255/58

C07F 9/40

C07F 9/54

C09K 11/06

H05B 33/14

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(22)Date of filing : 30.01.2001 (72)Inventor : ICHIMURA MARI

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(54) AMINOSTYRYLPHENANTHRENE COMPOUND AND INTERMEDIATE IN  
ITS SYNTHESIS, AND METHOD OF PRODUCTION THEREOF

(57)Abstract:

PROBLEM TO BE SOLVED: To provide an organic luminescent material which  
stably emits red light with high luminance, and a method of production thereof.

SOLUTION: The organic luminescent material comprises e.g. an  
aminostyrylphenanthrene compound represented by general formula (I)  
(wherein R2 is an unsubstituted aryl group; R1 is an aryl group which may have  
various substituents; and R3 to R5 are each H, a cyano group, a hydrocarbon  
group, or the like). It is produced by the condensation of a corresponding 4-(N,N-

diaryl amino)benzaldehyde with a phosphonic ester or phosphonium.

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LEGAL STATUS [Date of request for examination] 06.12.2004

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

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3.In the drawings, any words are not translated.

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## CLAIMS

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[Claim(s)]

[Claim 1] The amino styryl phenanthrene compound shown by the following general formula [I], [II], [III], [IV], or [V].

[Formula

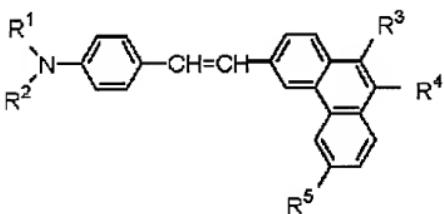
1]

General

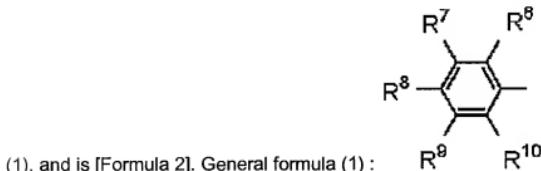
formula

[I]

:

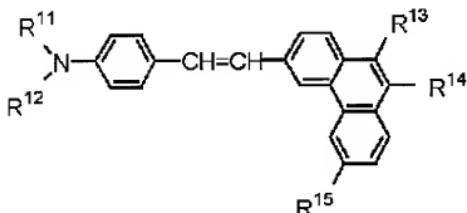


In [, however said general formula [I], it is the aryl group as which R2 is a non-permuted aryl group, and R1 is expressed in the following general formula

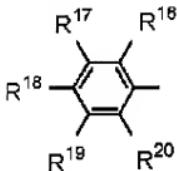


(However, in said general formula (1), R6, R7, R8, R9, and R10 are radicals which are identitas mutually or are different.) At least one The hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, it is the hydrocarbon amino group of with the hydrocarbon oxy-radical of with a carbon numbers of one or more saturation or partial saturation, and a carbon numbers of one or more saturation, or partial saturation, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom. R3 and R4 are radicals which are identitas mutually or are different. Those at least one A hydrogen atom, it is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, and R5 is the aryl group which may have the alkyl group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ]

[Formula 3] General formula [II] :



It is a radical which R11 and R12 are identitases mutually in [, however said general formula [II], or is different, is the aryl group expressed with the following general formula (2), and is [Formula 4]. General formula (2) :



(However, in said general formula (2), R16, R17, R18, R19, and R20 are radicals which are identitas mutually or are different.) At least one The hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, it is the hydrocarbon amino group of with the hydrocarbon oxy-radical of with a carbon numbers of one or more saturation or partial saturation, and a carbon numbers of one or more saturation, or partial saturation, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom. R13 and R14 are radicals which are identitas mutually or are different. Those at least one A hydrogen atom, it is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, and R15 is the aryl group which may have the alkyl group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ]

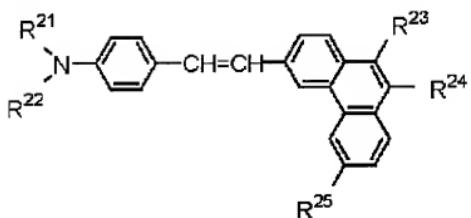
[Formula

5]

General

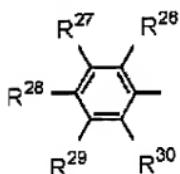
formula

[III]

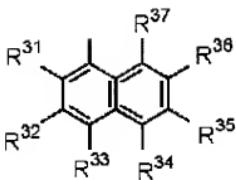


It is the aryl group as which R21 is expressed in the following general formula (3)

in [, however said general formula [III], and is [Formula 6]. General formula (3) :



In (said general formula (3 [ however), R26, R27, R28, R29, and R30 are radicals which are identitas mutually or are different, at least one is the hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, a with a carbon numbers of one or more hydrocarbon oxy-radical, the with a carbon numbers of one or more hydrocarbon amino group, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom.) and R22 are aryl groups expressed with the following general formula (4), and are [Formula 7] General formula (4) :



(However, in said general formula (4), R31, R32, R33, R34, R35, R36, and R37 are radicals which are identitas mutually or are different.) At least one The hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, it is the hydrocarbon amino group of with the hydrocarbon oxy-radical of with a carbon numbers of one or more saturation or partial saturation, and a carbon numbers of one or more saturation, or partial saturation, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom. R23 and R24 are radicals which are identitas mutually or are different. Those at least one A hydrogen atom, it is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, and R25 is the aryl group which may have the alkyl group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ]

[Formula

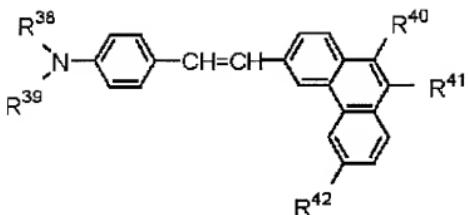
8]

General

formula

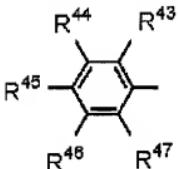
[IV]

:

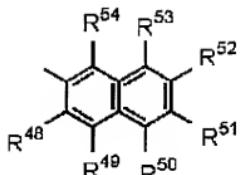


It is the aryl group as which R38 is expressed in the following general formula (5)

in [, however said general formula [IV], and is [Formula 9]. General formula (5) :



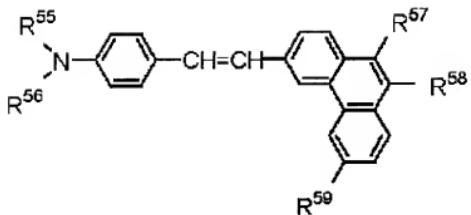
(However, in said general formula (5), R43, R44, R45, R46, and R47 are radicals which are identitas mutually or are different.) At least one The hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, it is the hydrocarbon amino group of with the hydrocarbon oxy-radical of with a carbon numbers of one or more saturation or partial saturation, and a carbon numbers of one or more saturation, or partial saturation, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom. R39 is an aryl group expressed with the following general formula (6), and



is [Formula 10]. General formula (6) :

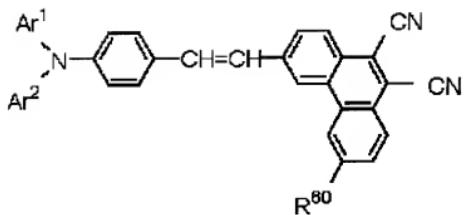
(However, in said general formula (6), R48, R49, R50, R51, R52, R53, and R54 are radicals which are identitas mutually or are different.) at least one is the hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, a hydrocarbon oxy-radical, the hydrocarbon amino group, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom. R40 and R41 are radicals which are identitas mutually or are different. Those at least one A hydrogen atom, it is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, and R42 is the aryl group which may have the alkyl group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ]

[Formula 11] General formula [V] :



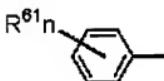
It is a radical which R55 and R56 are identitases mutually, or is different in [, however said general formula [V]. At least one is the hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation. R57 and R58 are radicals which are identitas mutually or are different. Those at least one A hydrogen atom, It is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, and R59 is the aryl group which may have the alkyl group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ] [Claim 2] The amino styryl phenanthrene compound which is expressed with the following general formula (7) and which was indicated to claim 1.

[Formula 12] General formula (7) :



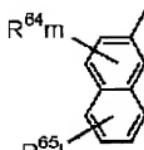
It is an aryl group in which Ar1 and Ar2 may have a substituent in  $\beta$ , however said general formula (7), respectively and which is identitas mutually or is different, is the radical chosen from the aryl group expressed with the following general formula (8), (9), (10), (11), (12), (13), (14), (15), and (16) when it has a

substituent, and is [Formula 13]. General formula (8) :

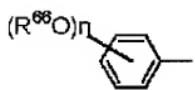


General formula (9):  $R^{62}m$  

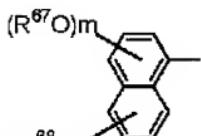
General formula (9) :



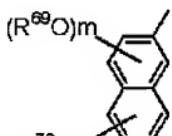
General formula (10) :  $R^{65}$



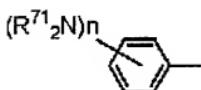
General formula (11) :



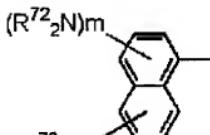
General formula (12) :  $(R^{68}O)_l$



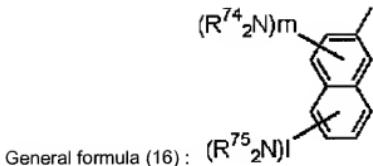
General formula (13) :  $(R^{70}O)_l$



General formula (14) :



General formula (15) :  $(R^{73}{}_2N)_l$

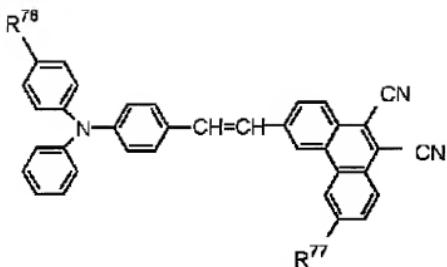


(However, it sets to said general formula (8), (9), (10), (11), (12), (13), (14), (15), and (16).) R61, R62, R63, R64, R65, R66, R67, R68, R69, R70, R71, R72, R73, R74, and R75 are the hydrocarbon groups of with identitas or a different carbon numbers of one or more saturation or partial saturation mutually. n is the integer of 0-5, m is the integer of 0-3, and I is the integer of 0-3. R60 is the aryl group which may have the alkyl group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ]

[Claim 3] The amino styryl phenanthrene compound whose carbon numbers which form said hydrocarbon group of R61, R62, R63, R64, R65, R66, R67, R68, R69, R70, R71, R72, R73, R74, and R75 are 1-6 and which was indicated to claim 2.

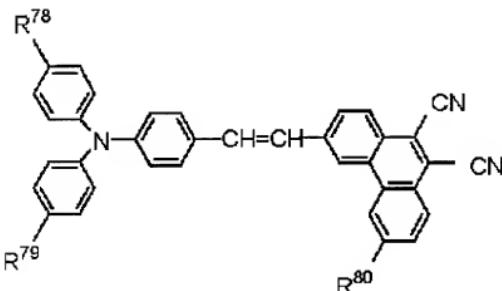
[Claim 4] The amino styryl phenanthrene compound which is expressed with the following general formula (17), (18), (19), (20), (21), (22), or (23) and which was indicated to claim 2.

[Formula 14] General formula (17) :



(However, in said general formula (17), R76 is the aryl group which may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R77 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

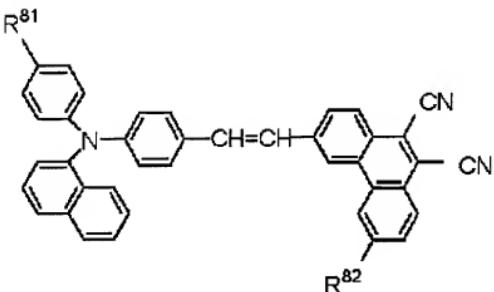
[Formula 15] General formula (18) :



(However, in said general formula (18), R78 and R79 are the aryl groups which

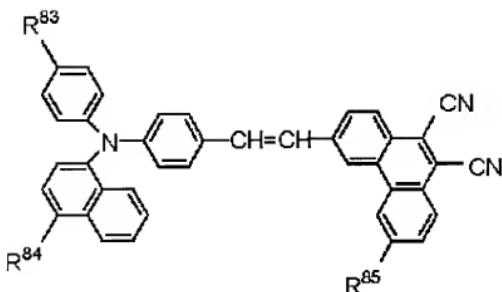
may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R80 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

[Formula 16] General formula (19) :



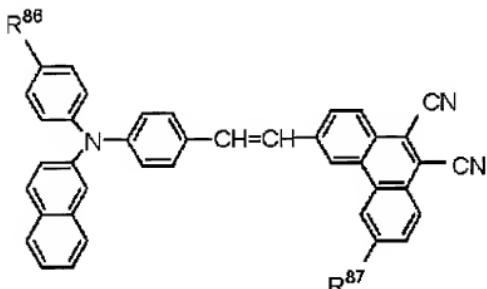
(However, in said general formula (19), R81 is the aryl group which may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R82 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

[Formula 17] General formula (20) :



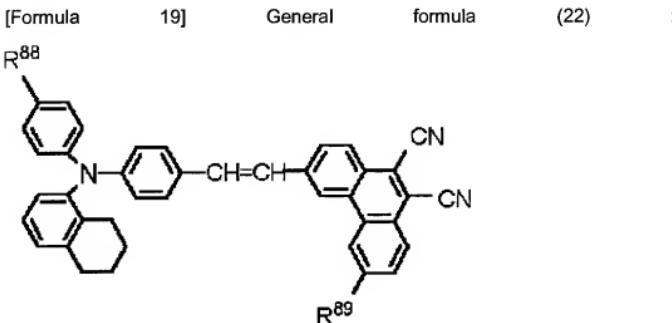
(However, in said general formula (20), R83 and R84 are the aryl groups which may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R85 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

[Formula 18] General formula (21) :



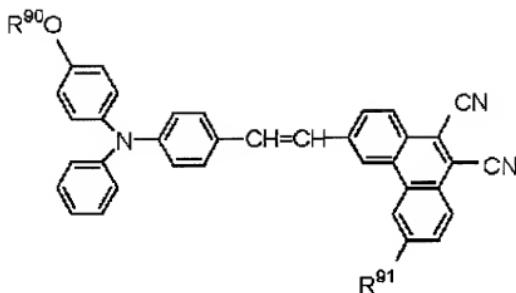
(However, in said general formula (21), R86 is the aryl group which may have

the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R87 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)



(However, in said general formula (22), R88 is the aryl group which may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R89 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

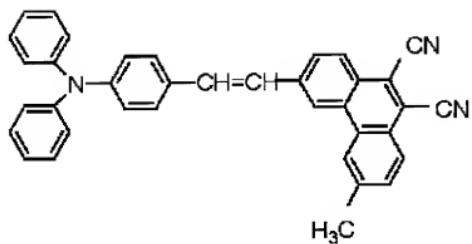




(However, in said general formula (23), R90 is the aryl group which may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R91 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

[Claim 5] following structure-expression (24)-1 and (24) -2, (24)-3, (24)-4, and (24) -5, (24)-6, (24)-7, and (24) -8, (24)-9, (24)-10, and (24) -- the amino styryl phenanthrene compound which is expressed with -11, (24)-12, and (24)-13 and which be indicated to claim 2.

[Formula 21] Structure expression (24) -1 :



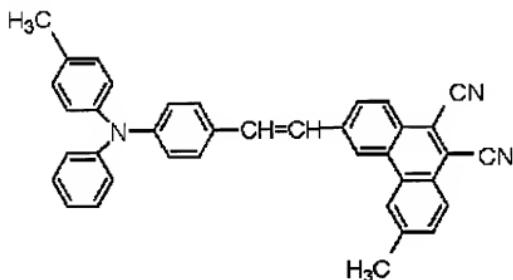
Structure

expression

(24)

-2

:



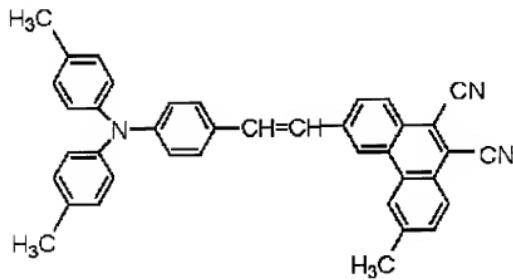
Structure

expression

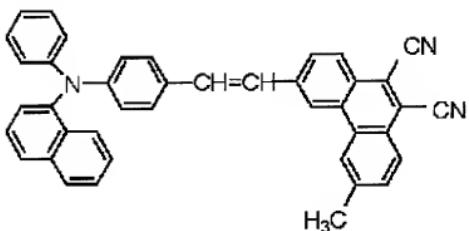
(24)

-3

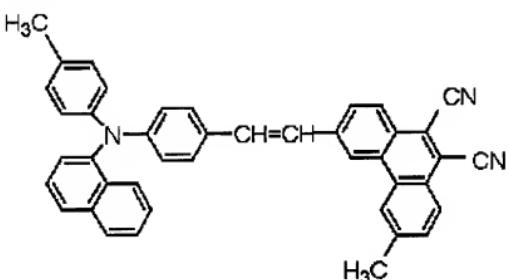
:



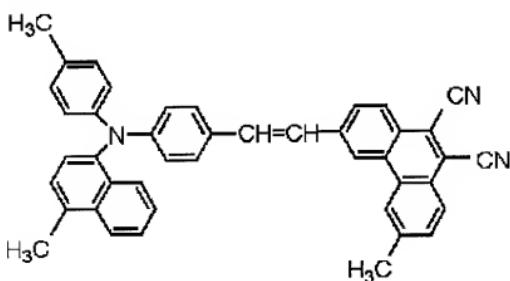
Structure expression (24) -4 :



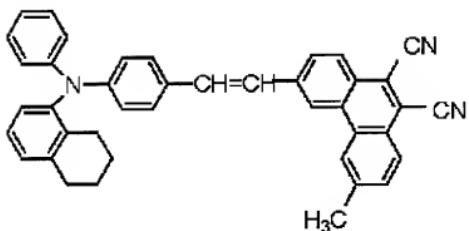
Structure expression (24) -5 :



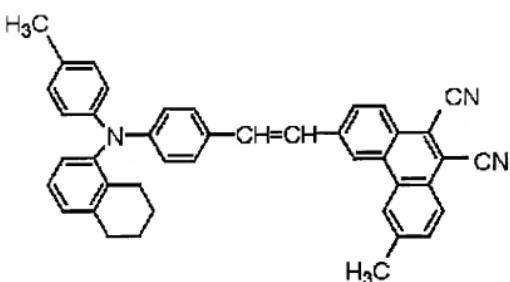
Structure expression (24) -6 :



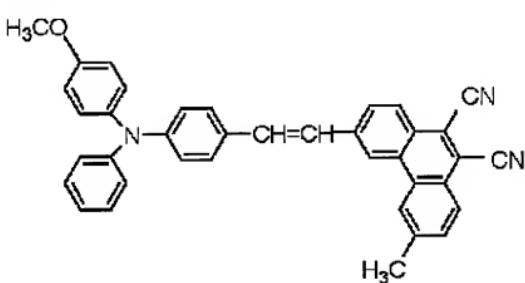
Structure expression (24) -7 :



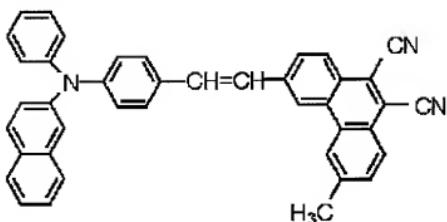
Structure expression (24) -8 :



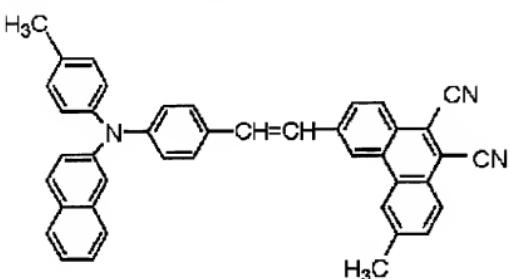
Structure expression (24) -9 :



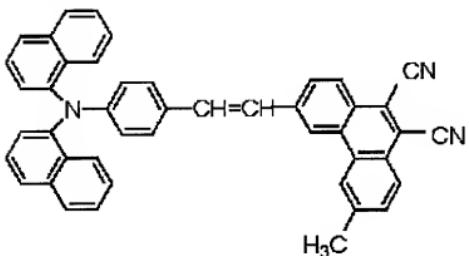
Structure expression (24) -10 :

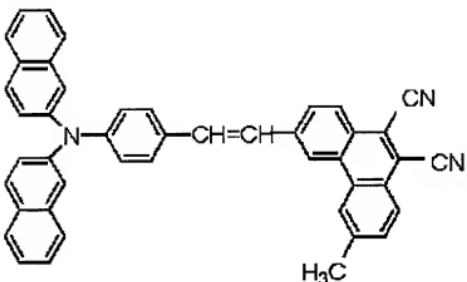


Structure expression (24) -11 :

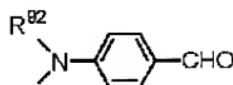


Structure expression (24) -12 :





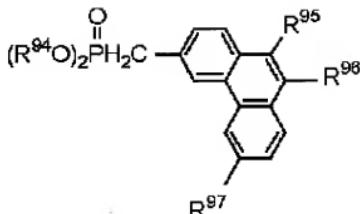
[Claim 6] The manufacture approach of an amino styryl phenanthrene compound of obtaining the amino styryl phenanthrene compound expressed with the following general formula [I], [II], [III], [IV], or [V] by carrying out condensation of the phosphonium and; which are expressed with the phosphonate and/or the following general formula [VIII] which are expressed with 4-(N and N-diaryl amino) benzaldehyde expressed with the following general formula [VI], and the; following general formula [VII].



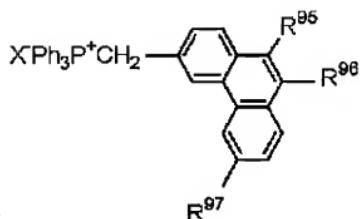
[Formula 22] General formula [VI] : R<sup>92</sup>-N(R<sup>93</sup>)-C(=O)-Phenyl

(However, in said general formula [VI], R<sup>92</sup> and R<sup>93</sup> are the aryl groups equivalent to following R1, R2, R11, R12, R21, R22, R38, R39 and R55, or R56,

respectively.)



[Formula 23] General formula [VII] :



General formula [VIII] :

(However, in said general formula [VII] and [VIII], R94 is a hydrocarbon group, R95 and R96 are the radicals equivalent to following R3, R4, R13, R14, R23, R24, R40, R41 and R57, or R58, respectively, R97 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent, and X is a halogen atom.)

[Formula

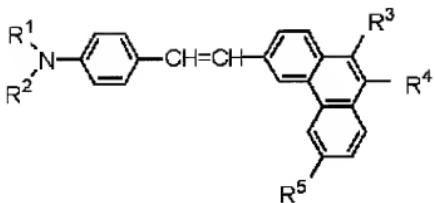
24]

General

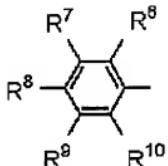
formula

[I]

:



In [, however said general formula [I], it is the aryl group as which R2 is a non-permuted aryl group, and R1 is expressed in the following general formula

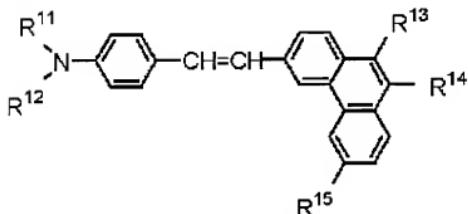


(1), and is [Formula 25]. General formula (1) :

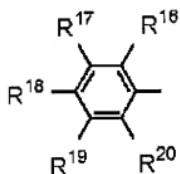
(However, in said general formula (1), R6, R7, R8, R9, and R10 are radicals which are identitas mutually or are different.) At least one The hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, it is the hydrocarbon amino group of with the hydrocarbon oxy-radical of with a carbon numbers of one or more saturation or partial saturation, and a carbon numbers of one or more saturation, or partial saturation, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom. R3 and R4 are radicals which are identitas mutually or are different. Those at least one A hydrogen atom, it is a cyano group, a nitro group, a trifluoromethyl

radical, or a halogen atom, the remainder is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, and R5 is the aryl group which may have the alkyl group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ]

[Formula 26] General formula [III] :



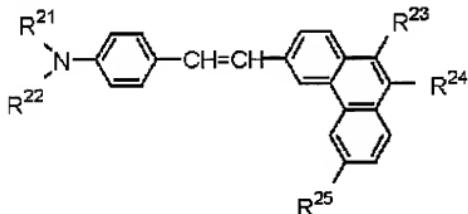
It is a radical which R11 and R12 are identitases mutually in [, however said general formula [III], or is different, is the aryl group expressed with the following general formula (2), and is [Formula 27]. General formula (2) :



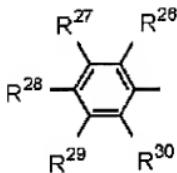
(However, in said general formula (2), R16, R17, R18, R19, and R20 are radicals which are identitas mutually or are different.) At least one The hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation

or partial saturation, it is the hydrocarbon amino group of with the hydrocarbon oxy-radical of with a carbon numbers of one or more saturation or partial saturation, and a carbon numbers of one or more saturation, or partial saturation, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom. R13 and R14 are radicals which are identitas mutually or are different. Those at least one A hydrogen atom, it is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, and R15 is the aryl group which may have the alkyl group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ]

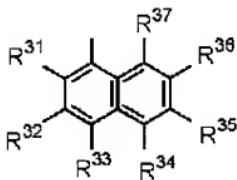
[Formula 28] General formula [III] :



It is the aryl group as which  $R^{21}$  is expressed in the following general formula (3) in [, however said general formula [III], and is [Formula 29]. General formula (3) :



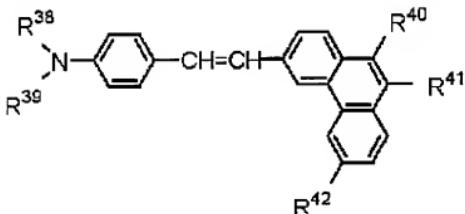
In (said general formula (3) [ however), R26, R27, R28, R29, and R30 are radicals which are identitas mutually or are different, at least one is the hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, a with a carbon numbers of one or more hydrocarbon oxy-radical, the with a carbon numbers of one or more hydrocarbon amino group, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom.) and R22 are aryl groups expressed with the following general formula (4), and are [Formula 30] General formula (4) :



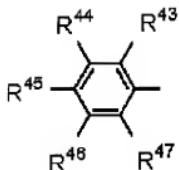
(However, in said general formula (4), R31, R32, R33, R34, R35, R36, and R37 are radicals which are identitas mutually or are different.) At least one The hydrocarbon group of with a hydrogen atom and a carbon numbers of one or

more saturation or partial saturation, it is the hydrocarbon amino group of with the hydrocarbon oxy-radical of with a carbon numbers of one or more saturation or partial saturation, and a carbon numbers of one or more saturation, or partial saturation, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom. R23 and R24 are radicals which are identitas mutually or are different. Those at least one A hydrogen atom, it is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, and R25 is the aryl group which may have the alkyl group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ]

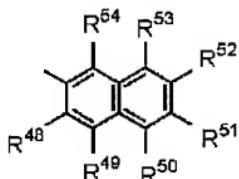
[Formula 31] General formula [IV] :



It is the aryl group as which R38 is expressed in the following general formula (5) in [, however said general formula [IV], and is [Formula 32]. General formula (5) :



(However, in said general formula (5), R43, R44, R45, R46, and R47 are radicals which are identitas mutually or are different.) At least one The hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, it is the hydrocarbon amino group of with the hydrocarbon oxy-radical of with a carbon numbers of one or more saturation or partial saturation, and a carbon numbers of one or more saturation, or partial saturation, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom. R39 is an aryl group expressed with the following general formula (6), and

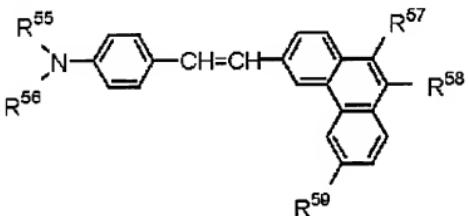


is [Formula 33]. General formula (6) :

(However, in said general formula (6), R48, R49, R50, R51, R52, R53, and R54 are radicals which are identitas mutually or are different.) at least one is the hydrocarbon group of with a hydrogen atom and a carbon numbers of one or

more saturation or partial saturation, a hydrocarbon oxy-radical, the hydrocarbon amino group, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom. R40 and R41 are radicals which are identitas mutually or are different. Those at least one A hydrogen atom, it is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, and R42 is the aryl group which may have the alkyl group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ]

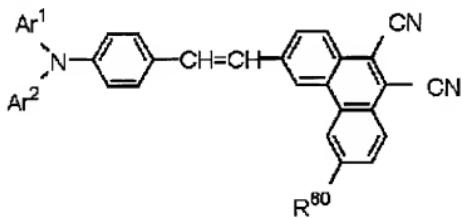
[Formula 34] General formula [V] :



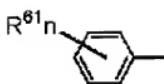
It is a radical which R55 and R56 are identitas mutually, or is different in [, however said general formula [V]. At least one is the hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation. R57 and R58 are radicals which are identitas mutually or are different. Those at least one A hydrogen atom, It is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a

nitro group, a trifluoromethyl radical, or a halogen atom, and R59 is the aryl group which may have the alkyl group with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ] [Claim 7] The manufacture approach of the amino styryl phenanthrene compound indicated to claim 6 to which the Wittig HONA (Wittig-Homer) reaction or the Wittig (Wittig) reaction performs said condensation, a carbanion is made to generate by processing said phosphonate and/or said phosphonium by the base in a solvent, and condensation of this carbanion and said 4-(N and N-diaryl amino) benzaldehyde is carried out.

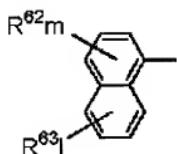
[Claim 8] It faces obtaining the amino styryl phenanthrene compound expressed with the following general formula (7), and is [Formula 35]. General formula (7) :



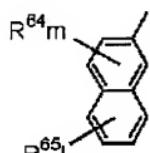
It is an aryl group in which Ar1 and Ar2 may have a substituent in [, however said general formula (7), respectively and which is identitas mutually or is different, is the radical chosen from the aryl group expressed with the following general formula (8), (9), (10), (11), (12), (13), (14), (15), and (16) when it has a



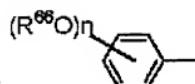
substituent, and is [Formula 36]. General formula (8) :



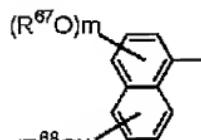
General formula (9) :



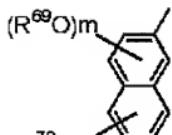
General formula (10) :



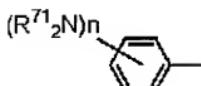
General formula (11) :



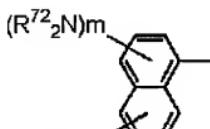
General formula (12) :



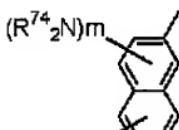
General formula (13) :  $(R^{69}O)m$   $(R^{70}O)l$



General formula (14) :



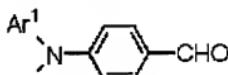
General formula (15) :  $(R^{72}N_2)m$   $(R^{73}N_2)l$



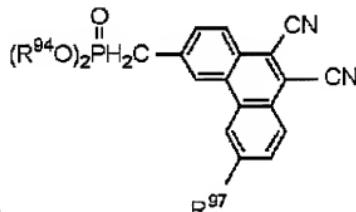
General formula (16) :  $(R^{74}N_2)m$   $(R^{75}N_2)l$

(However, it sets to said general formula (8), (9), (10), (11), (12), (13), (14), (15), and (16).) R61, R62, R63, R64, R65, R66, R67, R68, R69, R70, R71, R72, R73, R74, and R75 are the hydrocarbon groups of with identitas or a different carbon numbers of one or more saturation or partial saturation mutually. n is the integer of 0-5, m is the integer of 0-3, and l is the integer of 0-3. R60 is the aryl group

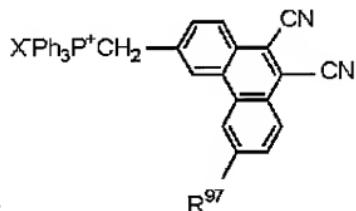
which may have the alkyl group with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ] The manufacture approach of the amino styryl phenanthrene compound indicated to claim 6 of carrying out condensation of; to the phosphonium expressed with the phosphonate and/or the following general formula (27) which are expressed with 4-(N and N-diaryl amino) benzaldehyde expressed with the following general formula (25), and the; following general formula (26).



[Formula 37] General formula (25) :  $Ar^2$



General formula (26) :



General formula (27) :

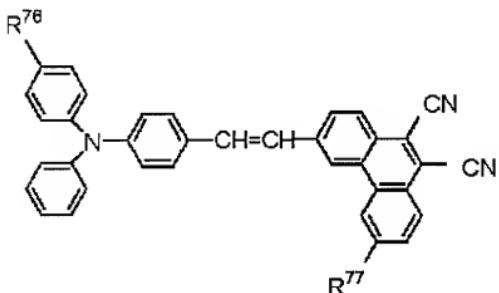
(However, in said general formula (25), (26), and (27), Ar1, Ar2, R94, R97, and X are the same as the above mentioned thing.)

[Claim 9] The manufacture approach of an amino styryl phenanthrene compound which makes said R94 the saturated hydrocarbon radical of carbon numbers 1-4 and which was indicated to claim 6.

[Claim 10] The manufacture approach of an amino styryl phenanthrene compound which sets to 1-6 the carbon number which forms said hydrocarbon of R61, R62, R63, R64, R65, R66, R67, R68, R69, R70, R71, R72, R73, R74, and R75 and which was indicated to claim 7.

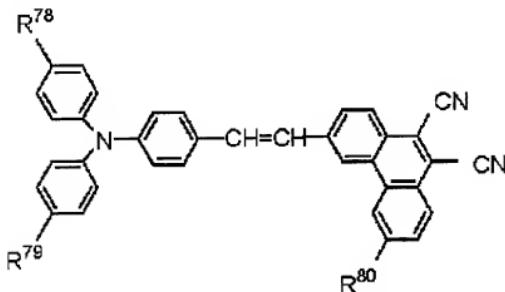
[Claim 11] The manufacture approach of the amino styryl phenanthrene compound indicated to claim 7 of obtaining the amino styryl phenanthrene compound expressed with the following general formula (17), (18), (19), (20), (21), (22), or (23).

[Formula 38] General formula (17) :



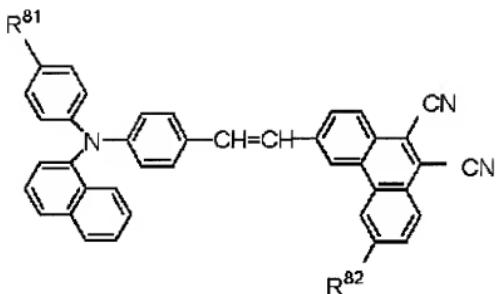
(However, in said general formula (17), R76 is the aryl group which may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R77 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

[Formula 39] General formula (18) :



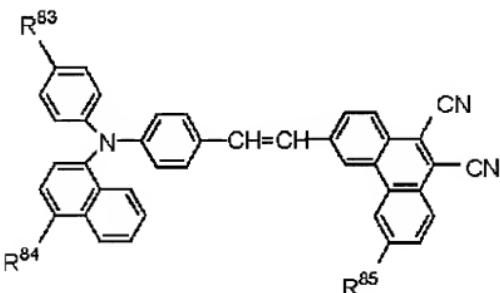
(However, in said general formula (18),  $\text{R}^{78}$  and  $\text{R}^{79}$  are the aryl groups which may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and  $\text{R}^{80}$  is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

[Formula 40] General formula (19) :



(However, in said general formula (19), R<sup>81</sup> is the aryl group which may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R<sup>82</sup> is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

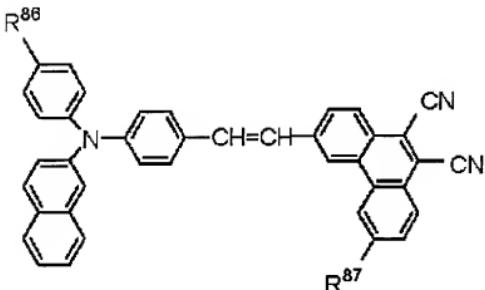
[Formula 41] General formula (20) :



(However, in said general formula (20), R<sup>83</sup> and R<sup>84</sup> are the aryl groups which

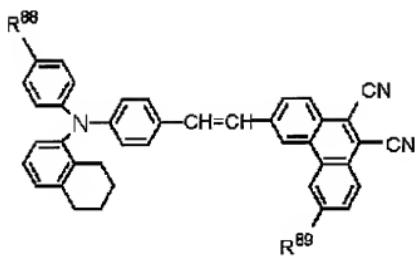
may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R85 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

[Formula 42] General formula (21) :



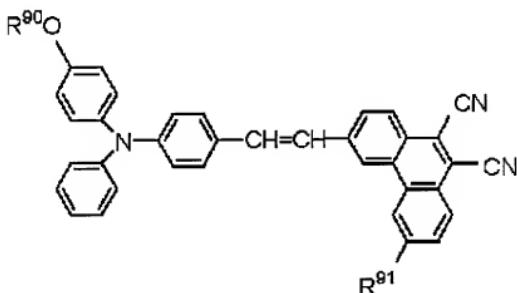
(However, in said general formula (21), R86 is the aryl group which may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R87 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

[Formula 43] General formula (22) :



(However, in said general formula (22), R88 is the aryl group which may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R89 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

[Formula 44] General formula (23) :

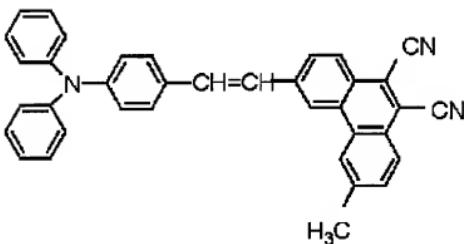


(However, in said general formula (23), R90 is the aryl group which may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a

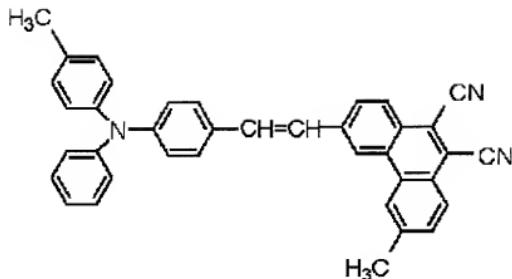
substituent, and R91 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

[Claim 12] The following structure expression (24) -1 and (24) -2, (24)-3, (24)-4, and (24) -5, (24)-6, (24)-7, and (24) -8, (24)-9, (24)-10, and (24) – the amino styryl phenanthrene compound expressed with -11, (24)-12, and (24)-13 The manufacture approach of the amino styryl phenanthrene compound indicated to claim 7 to acquire.

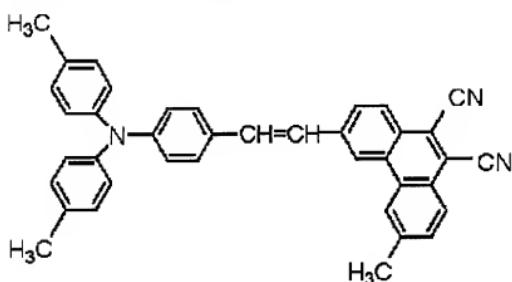
[Formula 45] Structure expression (24) -1 :



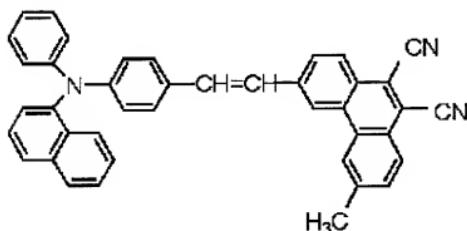
Structure expression (24) -2 :



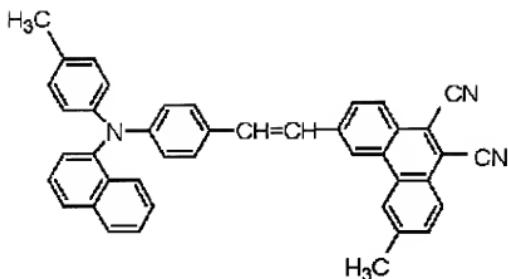
Structure expression (24) -3 :



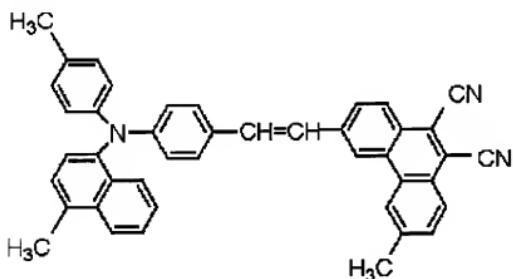
Structure expression (24) -4 :



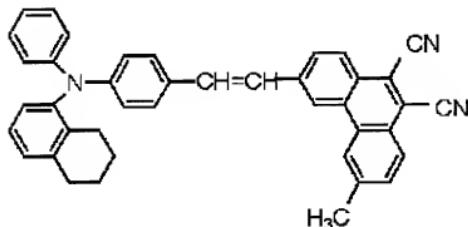
Structure expression (24) -5 :



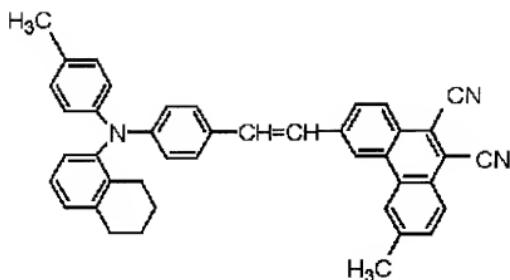
Structure expression (24) -6 :



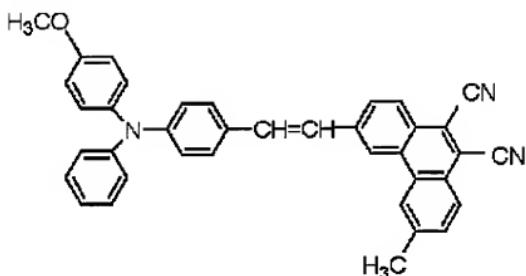
Structure expression (24) -7 :



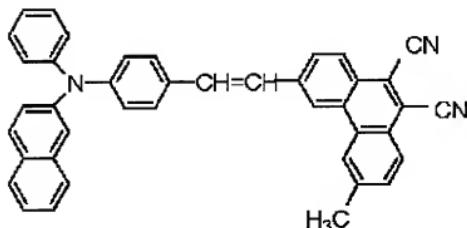
Structure expression (24) -8 :



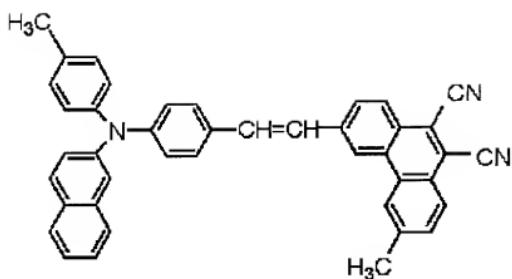
Structure expression (24) -9 :



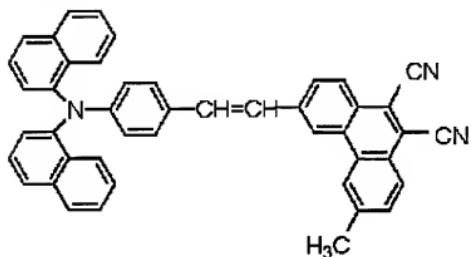
Structure expression (24) -10 :



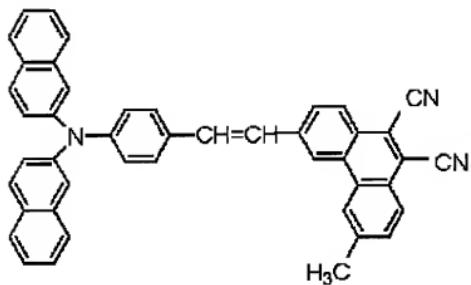
Structure expression (24) -11 :



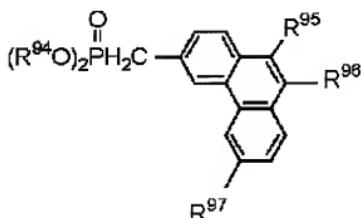
Structure	expression	(24)	-12	:
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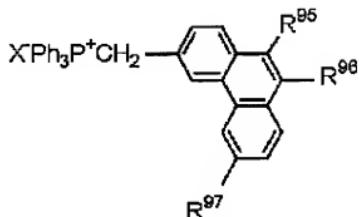
Structure	expression	(24)	-13	:
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[Claim 13] Phosphonate or phosphonium expressed with the following general formula [VII] or [VIII].



[Formula 46] General formula [VII] :



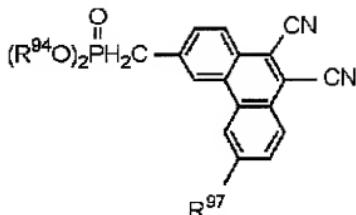
General formula [VIII] :

(However, in said general formula [VII] and [VIII], R<sup>94</sup> is a hydrocarbon group, R<sup>95</sup> and R<sup>96</sup> are the radicals equivalent to said R<sub>3</sub>, R<sub>4</sub>, R<sub>13</sub>, R<sub>14</sub>, R<sub>23</sub>, R<sub>24</sub>, R<sub>40</sub>, R<sub>41</sub>, R<sub>57</sub>, or R<sub>58</sub>, respectively, R<sup>97</sup> is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent, and X is a halogen atom.)

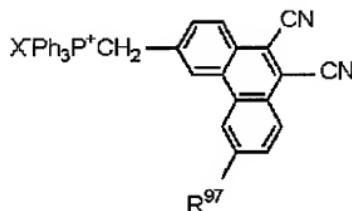
[Claim 14] Phosphonate or phosphonium said R<sup>94</sup> [ whose ] is the saturated hydrocarbon radical of carbon numbers 1-4 and which was indicated to claim 13.

[Claim 15] Phosphonate or phosphonium which is expressed with the following

general formula (26) and (27) and which was indicated to claim 13.



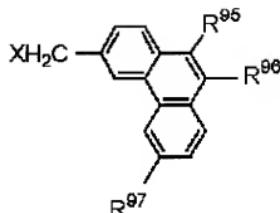
[Formula 47] General formula (26) :



General formula (27) :

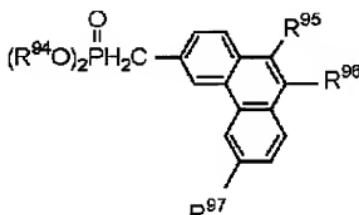
(However, in said general formula (26) and (27), R94, R97, and X are the same as the above mentioned thing.)

[Claim 16] The manufacture approach of of the phosphonate or phosphonium which obtains the phosphonate or phosphonium expressed with the following general formula [VII] or [VIII] by making the aryl halide compound expressed with the following general formula [IX], and the phosphorous acid trialkyl or triphenyl phosphine (PPh3) expressed with the following general formula [X] react.

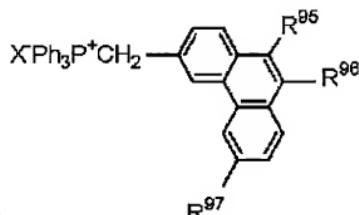


[Formula 48] General formula [IX] :

General formula [X] :P (OR94) 3 (however, in said general formula [X], R94 is a hydrocarbon group.)



[Formula 49] General formula [VII] :



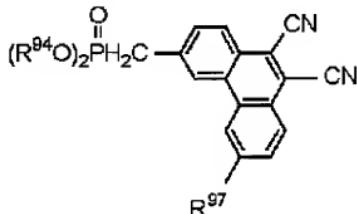
General formula [VIII] :

(However, in said general formula [VII] and [VIII], R94, R95, R96, R97, and X are the same as the above mentioned thing.)

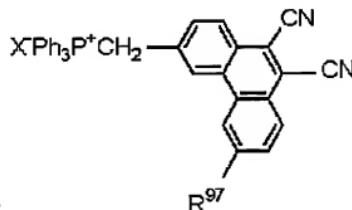
[Claim 17] The manufacture approach of of the phosphonate or phosphonium

which makes said R94 the saturated hydrocarbon radical of carbon numbers 1-4 and which was indicated to claim 16.

[Claim 18] The manufacture approach of of the phosphonate or phosphonium which obtains the phosphonate or phosphonium expressed with the following general formula (26) or (27) and which was indicated to claim 16.



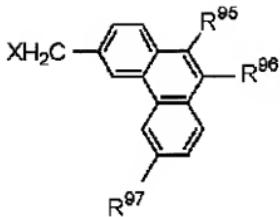
[Formula 50] General formula (26) :



General formula (27) :

(However, in said general formula (26) and (27), R94, R97, and X are the same as the above mentioned thing.)

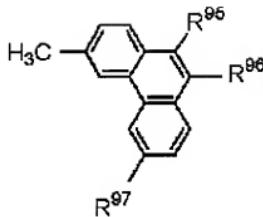
[Claim 19] The aryl halide compound expressed with the following general formula [IX].



[Formula 51] General formula [IX] :

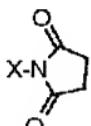
(However, in a general formula [IX],  $R^{95}$  and  $R^{96}$  are radicals which are identitas or are different, respectively, those at least one is a hydrogen atom, a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom,  $R^{97}$  is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent, and  $X$  is a halogen atom.)

[Claim 20] The manufacture approach of an aryl halide compound of obtaining the aryl halide compound expressed with the following general formula [IX] by making the phenanthrene compound expressed with the following general formula [XI], and N-halogenation succinimide expressed with a general formula [XII] reacting.



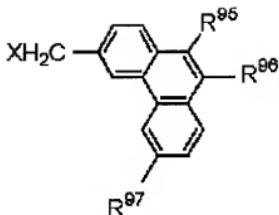
[Formula 52] General formula [XI] :

(However, in a general formula [XI], R95 and R96 are radicals which are identitas or are different, respectively, those at least one is a hydrogen atom, a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, and R97 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)



[Formula 53] General formula [XII] :

(However, in said general formula [XII], X is a halogen atom.)



[Formula 54] General formula [IX] :

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#### DETAILED DESCRIPTION

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##### [Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to these manufacture approaches at an amino styryl phenanthrene compound suitable as an organic luminescent material which presents red luminescence and its synthetic intermediate field, and a list.

[0002]

[Description of the Prior Art] It is spontaneous light, and a speed of response is high-speed, as one candidate of a flat-panel display without an angle-of-visibility dependency, organic electroluminescence devices (EL element) etc. attract attention one of these days, and the interest about an organic luminescent material is increasing as the component. Implementation of the full color organic

light emitting device which is in the place which can control the optical property of an ingredient to some extent by the molecular design, and created all of red, blue, and green three-primary-colors luminescence by each luminescent material by this is possible for the first advantage of an organic luminescent material.

[0003] Since the styryl compound shown by the following general formula [A] presents strong luminescence of blue - red to a visible-region field depending on the substituent introduced, it is available not only for an organic electroluminescence-devices ingredient but various applications. Furthermore, these ingredients are sublimability and have the advantage which can form the uniform amorphous film according to the process of vacuum deposition. Although the optical property of an ingredient can expect until to some extent by molecular orbital count etc. by the end of today, it cannot be overemphasized that the technique of manufacturing the ingredient demanded efficient in fact is the most important on industry.

[0004]

[Formula

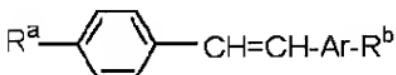
55]

General

formula

[A]

:



(However, in said general formula [A], Ar is the aryl group which may have a substituent, and Ra and Rb show the aryl group which may have the alkyl group of a hydrogen atom, saturation, or partial saturation, and a substituent, a cyano group, a halogen atom, a nitro group, a hydrocarbon oxy-radical, the hydrocarbon amino group, or a trifluoromethyl radical, respectively, and these may be the same or may differ.)

[0005]

[Problem(s) to be Solved by the Invention] Although many compounds which belong to said general formula [A] as an organic luminescent material have so far been manufactured, many are blue - green as luminescence of these ingredients is shown in JP,1-245087,A, JP,4-275268,A, JP,5-121168,A (above, Idemitsu Kosan, Inc.), JP,7-188649,A, JP,10-180582,A (above, Fuji Electric Co., Ltd.), etc. What presents luminescence of orange - red until now is [ only being reported slightly and ], and the efficient [manufacturing methods, such as Institute of Electronics, Information and Communication Engineers, technical research report, organic electronics, 17 and 7 (1992), Inorganic and Organic Electroluminescence 96 Berlin, and 101 (1996).] were not established, either.

[0006] The purpose of this invention is to offer a compound suitable as an organic luminescent material of the red which presents strong luminescence and its synthetic intermediate field, and the method of manufacturing these efficient

in view of the above present condition.

[0007]

[Means for Solving the Problem] In order that this invention may solve the above-mentioned technical problem, as a result of inquiring wholeheartedly, the amino styryl phenanthrene compound expressed with a general formula [I], [II], [III], [IV], or [V] presents strong luminescence, a header and its general and efficient manufacture approach are established, and this invention is reached [ that it can become a red luminescent material and ].

[0008] That is, this invention relates to the amino styryl phenanthrene compound (the compound of this invention is called hereafter.) expressed with the following general formula [I], [II], [III], [IV], or [V] first.

[Formula

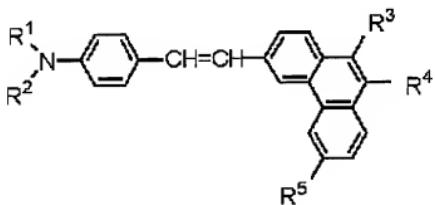
56]

General

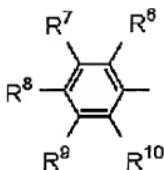
formula

[I]

:



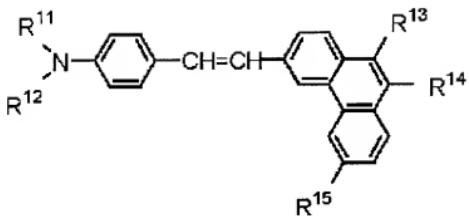
In [ , however said general formula [I], it is the aryl group as which R<sup>2</sup> is a non-permuted aryl group, and R<sup>1</sup> is expressed in the following general formula



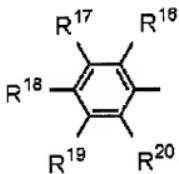
(1), and is [Formula 57]. General formula (1) :

(However, in said general formula (1), R6, R7, R8, R9, and R10 are radicals which are identitas mutually or are different.) At least one The hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, it is the hydrocarbon amino group of with the hydrocarbon oxy-radical of with a carbon numbers of one or more saturation or partial saturation, and a carbon numbers of one or more saturation, or partial saturation, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom. R3 and R4 are radicals which are identitas mutually or are different. Those at least one A hydrogen atom, it is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, and R5 is the aryl group which may have the alkyl group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ]

[Formula 58] General formula [II] :



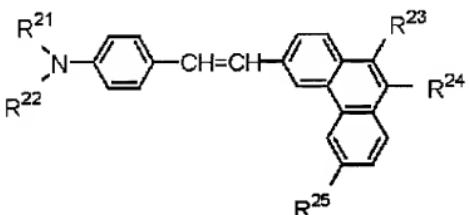
It is a radical which R11 and R12 are identitas mutually in [, however said general formula [II], or is different, is the aryl group expressed with the following general formula (2), and is [Formula 59]. General formula (2) :



(However, in said general formula (2), R16, R17, R18, R19, and R20 are radicals which are identitas mutually or are different.) At least one The hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, it is the hydrocarbon amino group of with the hydrocarbon oxy-radical of with a carbon numbers of one or more saturation or partial saturation, and a carbon numbers of one or more saturation, or partial saturation, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom. R13 and R14 are radicals which are identitas mutually or are different.

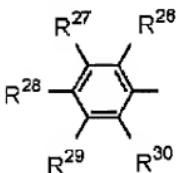
Those at least one A hydrogen atom, it is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, and R15 is the aryl group which may have the alkyl group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ]

[Formula 60] General formula [III] :



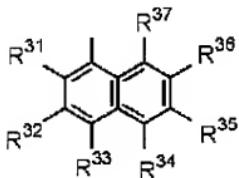
It is the aryl group as which R21 is expressed in the following general formula (3)

in [, however said general formula [III], and is [Formula 61]. General formula (3) :



In (said general formula (3 [ however), R26, R27, R28, R29, and R30 are radicals which are identitas mutually or are different, at least one is the hydrocarbon group of with a hydrogen atom and a carbon numbers of one or

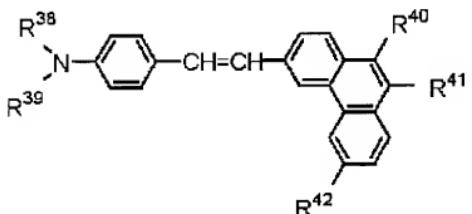
more saturation or partial saturation, a with a carbon numbers of one or more hydrocarbon oxy-radical, the with a carbon numbers of one or more hydrocarbon amino group, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom.) and R22 are aryl groups expressed with the following general formula (4), and are [Formula 62] General formula (4) :



(However, in said general formula (4), R31, R32, R33, R34, R35, R36, and R37 are radicals which are identitas mutually or are different.) At least one The hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, it is the hydrocarbon amino group of with the hydrocarbon oxy-radical of with a carbon numbers of one or more saturation or partial saturation, and a carbon numbers of one or more saturation, or partial saturation, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom. R23 and R24 are radicals which are identitas mutually or are different. Those at least one A hydrogen atom, it is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a

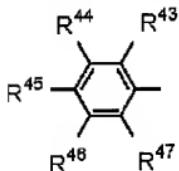
nitro group, a trifluoromethyl radical, or a halogen atom, and R25 is the aryl group which may have the alkyl group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ]

[Formula 63] General formula [IV] :



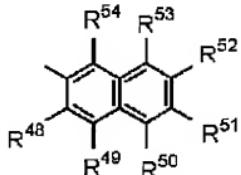
It is the aryl group as which R38 is expressed in the following general formula (5)

in [, however said general formula [IV], and is [Formula 64]. General formula (5) :



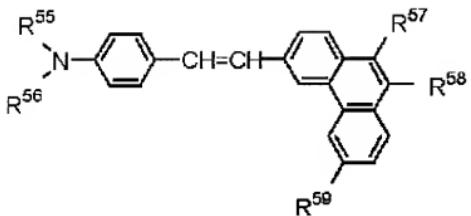
(However, in said general formula (5), R43, R44, R45, R46, and R47 are radicals which are identitas mutually or are different.) At least one The hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, it is the hydrocarbon amino group of with the hydrocarbon oxy-radical of with a carbon numbers of one or more saturation or partial

saturation, and a carbon numbers of one or more saturation, or partial saturation, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom. R39 is an aryl group expressed with the following general formula (6), and



is [Formula 65]. General formula (6) :

(However, in said general formula (6), R48, R49, R50, R51, R52, R53, and R54 are radicals which are identitas mutually or are different.) at least one is the hydrocarbon group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, a hydrocarbon oxy-radical, the hydrocarbon amino group, a trifluoromethyl radical, or a halogen atom, and the remainder is a hydrogen atom. R40 and R41 are radicals which are identitas mutually or are different. Those at least one A hydrogen atom, it is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, and R42 is the aryl group which may have the alkyl group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ]



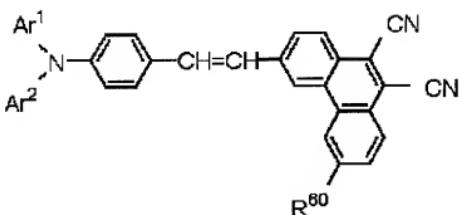
It is a radical which R55 and R56 are identitases mutually, or is different in [, however said general formula [V]. At least one is the hydrocarbon group of with a hydrogen atom or a carbon numbers of one or more saturation or partial saturation. R57 and R58 are radicals which are identitas mutually or are different. Those at least one A hydrogen atom, it is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, and R59 is the aryl group which may have the alkyl group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent. ] [0009]

It is the compound which can use the compound of this invention effectively as an organic luminescent material which shows red luminescence, and has the high melting point, and when excelled in electric, thermal, or chemical stability, it is amorphous, and since a vitreous state can be formed in preparation, vacuum evaporationo etc. can be performed.

[0010] As for the compound of this invention, what is expressed with the

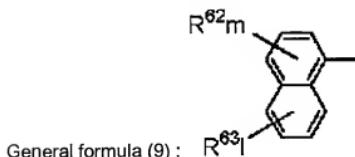
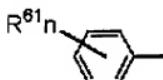
following general formula is desirable.

[Formula 67] General formula (7) :

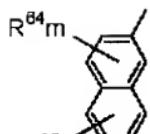


It is an aryl group in which Ar1 and Ar2 may have a substituent in [, however said general formula (7), respectively and which is identitas mutually or is different, is the radical chosen from the aryl group expressed with the following general formula (8), (9), (10), (11), (12), (13), (14), (15), and (16) when it has a

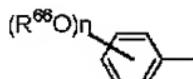
substituent, and is [Formula 68]. General formula (8) :



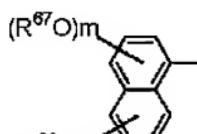
General formula (9) :



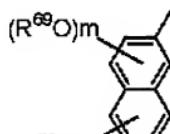
General formula (10) :  $R^{64}I$



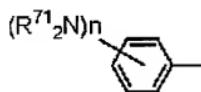
General formula (11) :



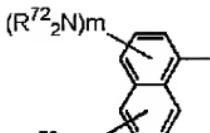
General formula (12) :  $(R^{68}O)I$



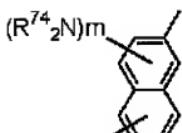
General formula (13) :  $(R^{70}O)I$



General formula (14) :



General formula (15) :  $(R^{73}N)_l$

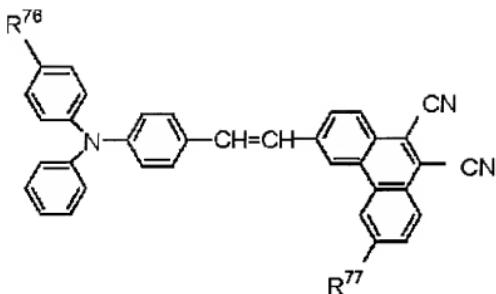


General formula (16) :  $(R^{75}N)_l$

(However, it sets to said general formula (8), (9), (10), (11), (12), (13), (14), (15), and (16).) R61, R62, R63, R64, R65, R66, R67, R68, R69, R70, R71, R72, R73, R74, and R75 are the hydrocarbon groups of with a carbon numbers of one or more (preferably 1-6) which are identitas mutually or are different saturation, or partial saturation. n is the integer of 0-5, m is the integer of 0-3, and l is the integer of 0-3. R60 is the aryl group which may have the alkyl group of with a hydrogen atom and a carbon numbers of one or more saturation or partial saturation, or a substituent.]

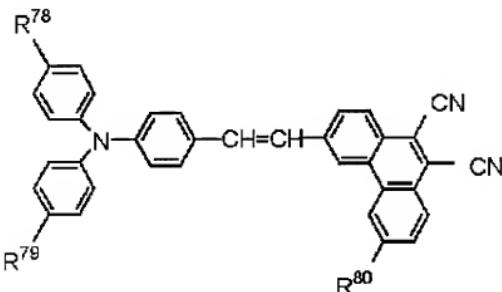
[0011] As for the compound of this invention, more specifically, what is expressed with the following general formula (17), (18), (19), (20), (21), (22), or (23) is good.

[Formula 69] General formula (17) :



(However, in said general formula (17), R<sup>76</sup> is the aryl group which may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R<sup>77</sup> is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

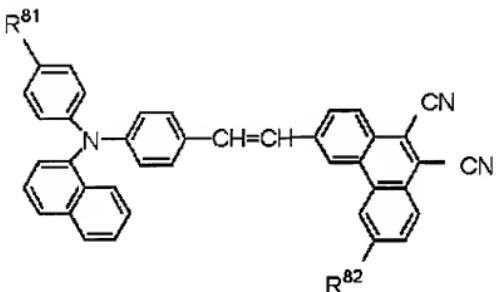
[Formula 70] General formula (18) :



(However, in said general formula (18), R<sup>78</sup> and R<sup>79</sup> are the aryl groups which

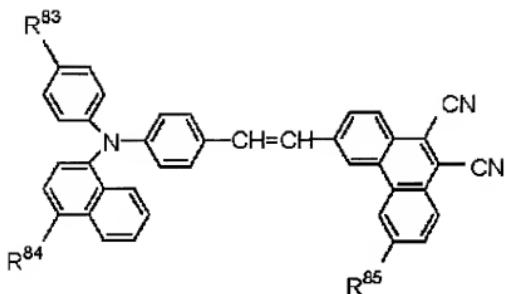
may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R80 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

[Formula 71] General formula (19) :



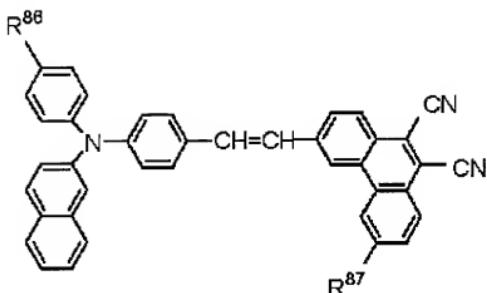
(However, in said general formula (19),  $\text{R}^{81}$  is the aryl group which may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and  $\text{R}^{82}$  is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

[Formula 72] General formula (20) :



(However, in said general formula (20), R83 and R84 are the aryl groups which may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R85 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

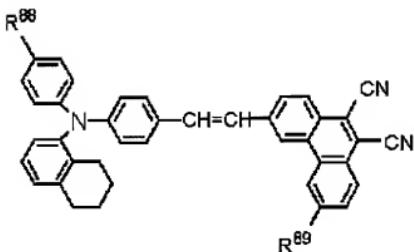
[Formula 73] General formula (21) :



(However, in said general formula (21), R86 is the aryl group which may have

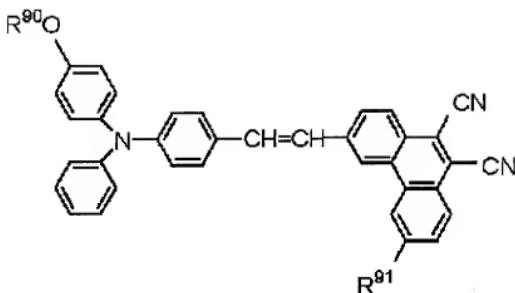
the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R87 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

[Formula 74] General formula (22) :



(However, in said general formula (22), R88 is the aryl group which may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R89 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

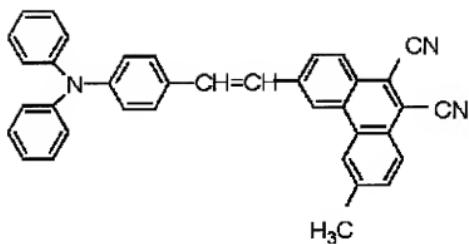
[Formula 75] General formula (23) :



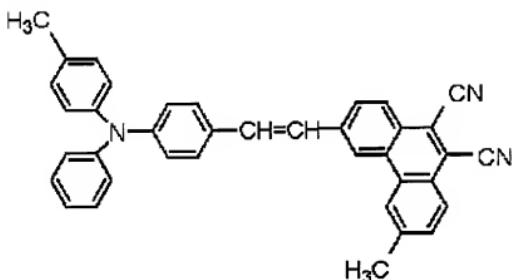
(However, in said general formula (23), R90 is the aryl group which may have the alkyl group of the saturation of carbon numbers 1-6, or partial saturation, or a substituent, and R91 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

[0012] the compound of this invention -- following structure-expression (24)-1 and (24)-2, (24)-3, (24)-4, and (24)-5, (24)-6, (24)-7, and (24)-8, (24)-9, (24)-10, and (24) -- what is expressed with -11, (24)-12, and (24)-13 is illustrated concretely.

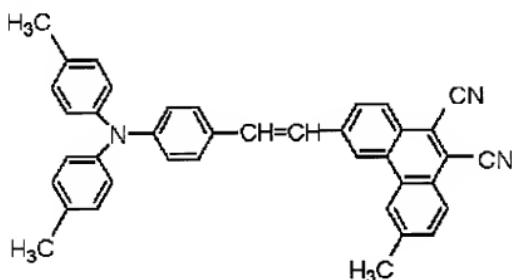
[Formula 76] Structure expression (24) -1 :



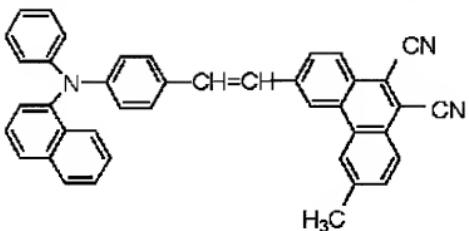
Structure expression (24) -2 :



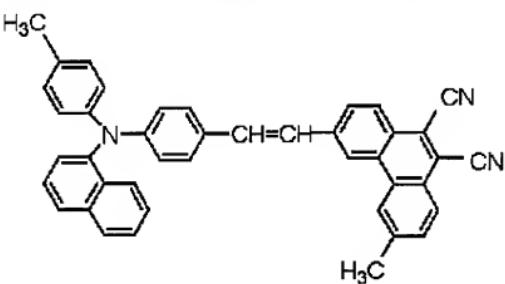
Structure expression (24) -3 :



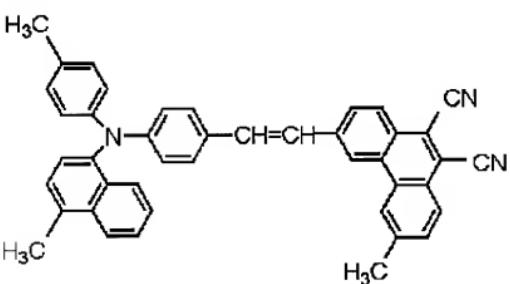
Structure expression (24) -4 :



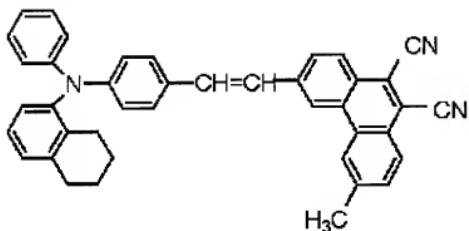
Structure expression (24) -5 :



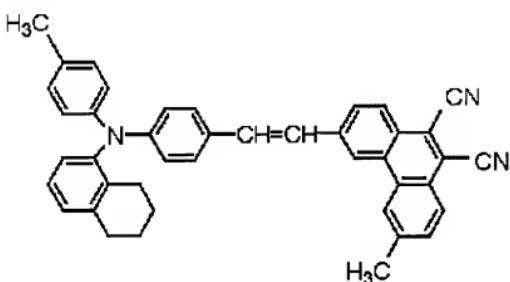
Structure expression (24) -6 :



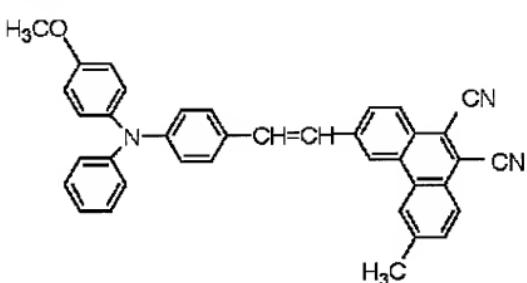
Structure expression (24) -7 :



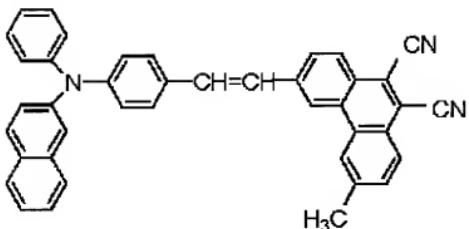
Structure expression (24) -8 :



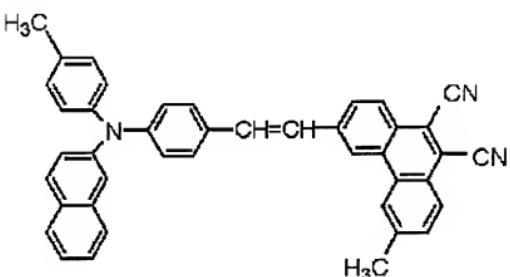
Structure expression (24) -9 :



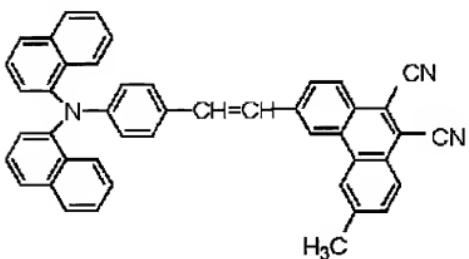
Structure expression (24) -10 :



Structure expression (24) -11 :



Structure expression (24) -12 :



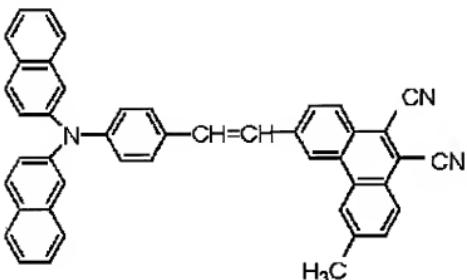
Structure

expression

(24)

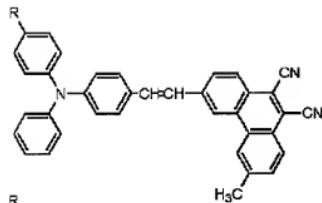
-13

:

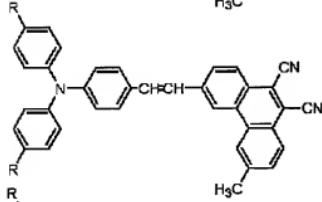


[0013] The following compound can also be illustrated besides these.

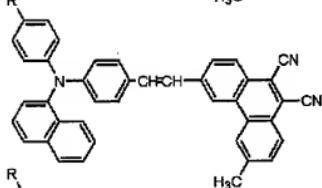
[Formula 77]



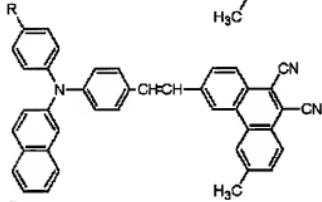
R=C<sub>2</sub>H<sub>5</sub>, *t*-C<sub>3</sub>H<sub>7</sub>, *i*-C<sub>4</sub>H<sub>9</sub>, *t*-C<sub>4</sub>H<sub>9</sub>,  
*cyclo*-C<sub>6</sub>H<sub>10</sub>, C<sub>6</sub>H<sub>5</sub>



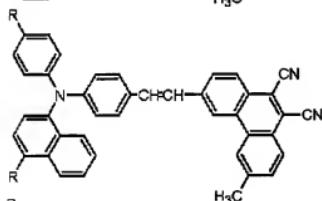
R=C<sub>2</sub>H<sub>5</sub>, *t*-C<sub>3</sub>H<sub>7</sub>, *i*-C<sub>4</sub>H<sub>9</sub>, *t*-C<sub>4</sub>H<sub>9</sub>,  
*cyclo*-C<sub>6</sub>H<sub>10</sub>, C<sub>6</sub>H<sub>5</sub>



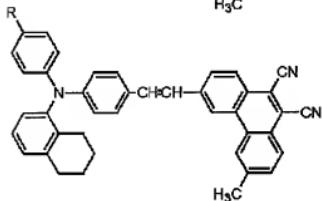
R=C<sub>2</sub>H<sub>5</sub>, *i*-C<sub>3</sub>H<sub>7</sub>, *i*-C<sub>4</sub>H<sub>9</sub>, *t*-C<sub>4</sub>H<sub>9</sub>,  
*cyclo*-C<sub>6</sub>H<sub>10</sub>, C<sub>6</sub>H<sub>5</sub>



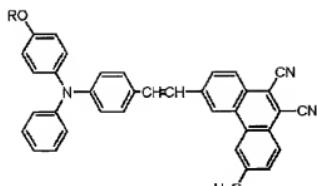
R=C<sub>2</sub>H<sub>5</sub>, *i*-C<sub>3</sub>H<sub>7</sub>, *i*-C<sub>4</sub>H<sub>9</sub>, *t*-C<sub>4</sub>H<sub>9</sub>,  
*cyclo*-C<sub>6</sub>H<sub>10</sub>, C<sub>6</sub>H<sub>5</sub>



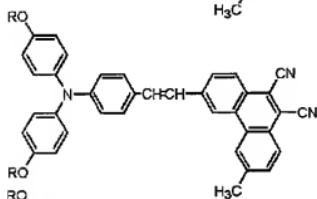
R=C<sub>2</sub>H<sub>5</sub>, *i*-C<sub>3</sub>H<sub>7</sub>, *i*-C<sub>4</sub>H<sub>9</sub>, *t*-C<sub>4</sub>H<sub>9</sub>,  
*cyclo*-C<sub>6</sub>H<sub>10</sub>, C<sub>6</sub>H<sub>5</sub>



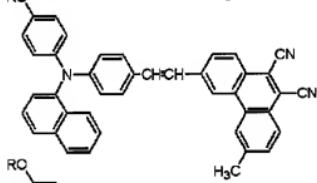
R=C<sub>2</sub>H<sub>5</sub>, *i*-C<sub>3</sub>H<sub>7</sub>, *i*-C<sub>4</sub>H<sub>9</sub>, *t*-C<sub>4</sub>H<sub>9</sub>,  
*cyclo*-C<sub>6</sub>H<sub>10</sub>, C<sub>6</sub>H<sub>5</sub>



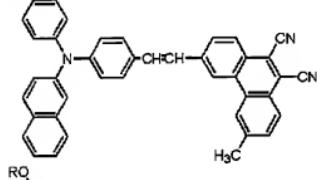
R=C<sub>2</sub>H<sub>5</sub>, *i*-C<sub>3</sub>H<sub>7</sub>, *i*-C<sub>4</sub>H<sub>9</sub>, *t*-C<sub>4</sub>H<sub>9</sub>,  
*cyclo*-C<sub>6</sub>H<sub>10</sub>, C<sub>6</sub>H<sub>5</sub>



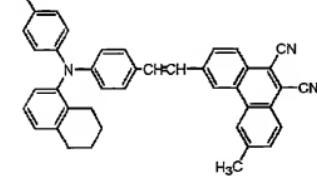
R=CH<sub>3</sub>, C<sub>2</sub>H<sub>5</sub>, *i*-C<sub>3</sub>H<sub>7</sub>, *i*-C<sub>4</sub>H<sub>9</sub>, *t*-C<sub>4</sub>H<sub>9</sub>,  
*cyclo*-C<sub>6</sub>H<sub>10</sub>, C<sub>6</sub>H<sub>5</sub>



R=CH<sub>3</sub>, C<sub>2</sub>H<sub>5</sub>, *i*-C<sub>3</sub>H<sub>7</sub>, *i*-C<sub>4</sub>H<sub>9</sub>, *t*-C<sub>4</sub>H<sub>9</sub>,  
*cyclo*-C<sub>6</sub>H<sub>10</sub>, C<sub>6</sub>H<sub>5</sub>



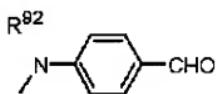
R=CH<sub>3</sub>, C<sub>2</sub>H<sub>5</sub>, *i*-C<sub>3</sub>H<sub>7</sub>, *i*-C<sub>4</sub>H<sub>9</sub>, *t*-C<sub>4</sub>H<sub>9</sub>,  
*cyclo*-C<sub>6</sub>H<sub>10</sub>, C<sub>6</sub>H<sub>5</sub>



R=CH<sub>3</sub>, C<sub>2</sub>H<sub>5</sub>, *i*-C<sub>3</sub>H<sub>7</sub>, *t*-C<sub>4</sub>H<sub>9</sub>, *t*-C<sub>4</sub>H<sub>9</sub>,  
*cyclo*-C<sub>6</sub>H<sub>10</sub>, C<sub>6</sub>H<sub>5</sub>

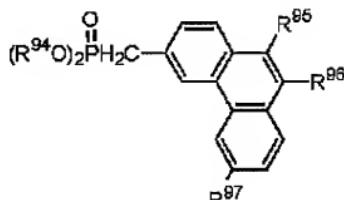
[0014] This invention as an approach of manufacturing the compound of this invention efficient again By carrying out condensation of the phosphonium and;

which are expressed with the phosphonate and/or the following general formula [VIII] which are expressed with the amino benzaldehyde expressed with the following general formula [VI], and the following general formula [VII]. The manufacture approach of this invention of obtaining the amino styryl phenanthrene compound shown by said general formula [I], [II], [III], [IV], or [V] is also offered.

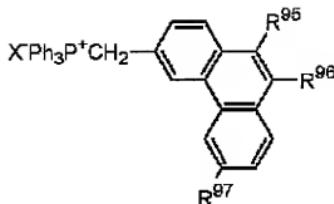


[Formula 78] General formula [VI] : R<sup>92</sup>  
R<sup>93</sup>

(However, in said general formula [VI], R<sup>92</sup> and R<sup>93</sup> are the aryl groups equivalent to said R<sub>1</sub>, R<sub>2</sub>, R<sub>11</sub>, R<sub>12</sub>, R<sub>21</sub>, R<sub>22</sub>, R<sub>38</sub>, R<sub>39</sub>, R<sub>55</sub>, or R<sub>56</sub>, respectively.)



[Formula 79] General formula [VII] :

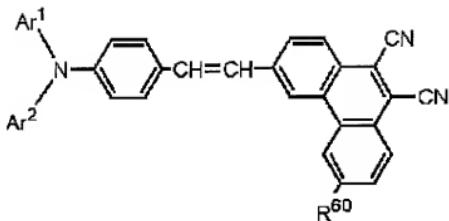


General formula [VIII] :

(However, in said general formula [VII] and [VIII], R94 is a hydrocarbon group (preferably saturated hydrocarbon radical of carbon numbers 1-4).) R95 and R96 are the radicals equivalent to said R3, R4, R13, R14, R23, R24, R40, R41, R57, or R58, respectively. R97 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent, and X is a halogen atom.

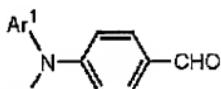
[0015] The manufacture approach of the compound of this invention performs said condensation by the Wittig HONA (Wittig-Horner) reaction or the Wittig (Wittig) reaction, by processing said phosphonate and/or said phosphonium by the base in a solvent, makes a carbanion generate and, specifically, carries out condensation to this carbanion and said 4-(N and N-diaryl amino) benzaldehyde.

[0016] For example, it faces obtaining the amino styryl phenanthrene compound expressed with the following general formula (7), and is [Formula 80]. General

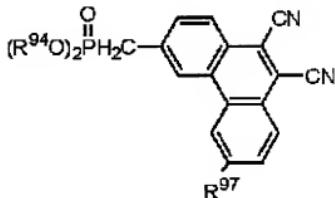


formula (7);

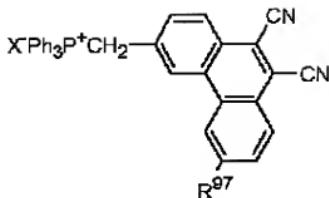
In (said general formula (7 [ however, ]), Ar1, Ar2, and R60 are the same as the above mentioned thing respectively. Condensation of the phosphonium and; which are expressed with 4-(N and N-diaryl amino) benzaldehyde, the phosphonate of the; following general formula (23), and/or the following general formula (24) which are expressed with) and the following general formula (25) is carried out.



[Formula 81] General formula (25) :  $Ar^2$



General formula (26) :

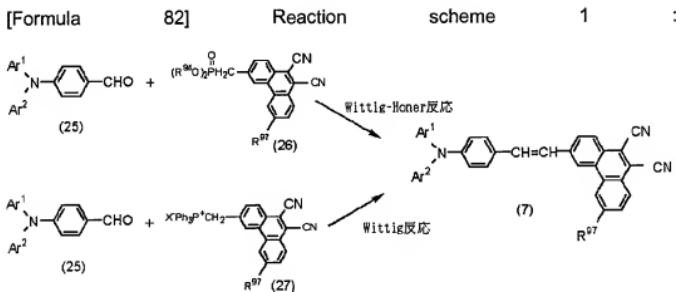


General formula (27) :

(However, in said general formula (26) and (27), R94, R97, and X are the same

as the above mentioned thing.)

[0017] If this reaction is expressed with a scheme, it will become, for example like the reaction scheme 1.



[0018] First, by processing the compound of said general formula (26) or said general formula (27) with a base in a suitable solvent, this reaction begins from generating a carbanion and is completed by next condensing this carbanion with the aldehyde of said general formula (25). The following can be considered as a

combination of a base and a solvent.

[0019] A sodium hydroxide/water, a sodium carbonate/water, potassium carbonate/water, a sodium ethoxide/ethanol, or dimethylformamide, Sodium methoxide / methanol-diethylether mixed solvent, or dimethylformamide, Triethylamine / ethanol, a jig lime, chloroform, or nitromethane, A pyridine / methylene chloride or nitromethane, 1, 5-JISAZA bicyclo [4.3.0] non-5-en / dimethyl sulfoxide, Potassium t-butoxide / dimethyl sulfoxide, a tetrahydrofuran, benzene, or dimethylformamide, A phenyl lithium / diethylether or a tetrahydrofuran, t-butyl lithium / diethylether, or a tetrahydrofuran, Sodium amide/ammonia, sodium hydride / dimethylformamide or a tetrahydrofuran, triethyl sodium / diethylether, or a tetrahydrofuran.

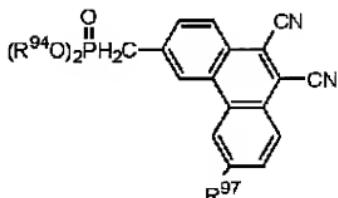
[0020] This reaction advances comparatively at low temperature (-30 degrees C - 30 degrees C), and in addition to purification of the specified substance by the chromatography being easy since it is alternative, since the compound of this invention of said general formula (7) has high crystallinity, it can raise purity with recrystallization. Although not asked especially about the approach of recrystallization, in the approach of dissolving in an acetone and adding a hexane, or toluene, the heating dissolution is carried out and the approach of condensing and cooling is simple. Ordinary pressure may perform this reaction in 3 - 24 hours.

[0021] By the manufacture approach of the compound of this invention, said general formula (17), (18), The amino styryl phenanthrene compound expressed with (19), (20), (21), (22), or (23) can be obtained. Specifically Said general formula (24) -1 and (24) -2, (24)-3, (24)-4, and (24) -5, (24)-6, (24)-7, and (24) -8, (24)-9, (24)-10, and (24) -- the amino styryl phenanthrene compound expressed with -11, (24)-12, or (24)-13 It can obtain.

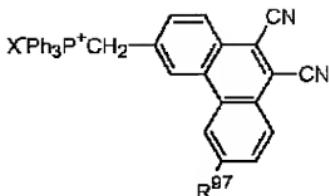
[0022] This invention also offers various compounds suitable as synthetic intermediate field of the compound of this invention again.

[0023] That is, they are the phosphonate expressed with said general formula [VII] used as synthetic intermediate field of the amino styryl phenanthrene compound expressed with said general formula [I], [II], [III], [IV], or [V], or said general formula [VIII] phosphonium.

[0024] This synthetic intermediate field (the synthetic intermediate field 1 of this invention are called hereafter.) are specifically expressed with the following general formula (26) or (27).



[Formula 83] General formula (26) :

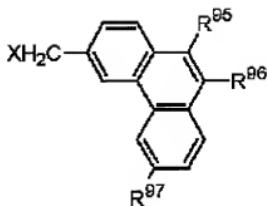


General formula (27) :

(However, in said general formula (26) and (27), R94, R97, and X are the same as the above mentioned thing.)

[0025] The synthetic intermediate field of this invention can be drawn as follows from the synthetic intermediate field as the precursor.

[0026] The phosphonium expressed with the phosphonate or the following general formula [VIII] expressed with the following general formula [VII] is obtained as synthetic intermediate field by making the aryl halide compound expressed with the following general formula [IX], and the phosphite trialkyl or triphenyl phosphine (PPh<sub>3</sub>) expressed with the following general formula [X] react. This reaction is good as 30 minutes - reaction-time 24 hours at the reaction temperature of 120 degrees C - 160 degrees C, and ordinary pressure in solvents, such as a xylene which has a non-solvent or the boiling point 120 degrees C or more, or the phosphite trialkyl of an overlarge.



[Formula 84] General formula [IX] :

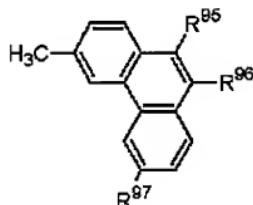
(However, in a general formula [IX], R95 and R96 are radicals which are identitas or are different, respectively, those at least one is a hydrogen atom, a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, the remainder is a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, R97 is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent, and X is a halogen atom.)

General formula [X] :P (OR94) 3 (however, in said general formula [X], R94 is the hydrocarbon group of the saturation of a hydrocarbon group, especially carbon numbers 1-4, or partial saturation.) [0027] This invention also offers the aryl halide compound (the synthetic intermediate field 2 of this invention are called hereafter.) expressed with said general formula [IX] as synthetic intermediate field for obtaining the synthetic intermediate field 1 again.

[0028] The synthetic intermediate product 2 of this invention can be obtained by making the dimethyl phenanthrene compound expressed with the following

general formula [XI], and N-halogenation succinimide expressed with the following general formula [XII] react to the bottom of an optical exposure. For example, it is made to react by the reaction time of 30 - 48 hours by the temperature of 20-120 degrees C, and ordinary pressure among solvents, such as a carbon tetrachloride, chloroform, benzene, and a chlorobenzene, using the light source of a high pressure mercury vapor lamp, a low pressure mercury lamp, a xenon LGT, a halogen LGT, daylight, a fluorescent lamp, etc.

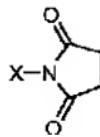
[0029]



[Formula 85] General formula [XI] :

(However, in a general formula [XI], R<sup>95</sup> and R<sup>96</sup> are radicals which are identitas or are different, respectively, those at least one is a hydrogen atom, a cyano group, a nitro group, a trifluoromethyl radical, or a halogen atom, and R<sup>97</sup> is the aryl group which may have the alkyl group of the saturation of a hydrogen atom and carbon numbers 1-6, or partial saturation, or a substituent.)

[0030]



[Formula 86] General formula [XII] :

[(However, in said general formula [XII], X is a halogen atom.) 0031] The following reaction scheme 2 can show the reaction which obtains each synthetic intermediate fields 1 and 2 described above, respectively.

[0032]

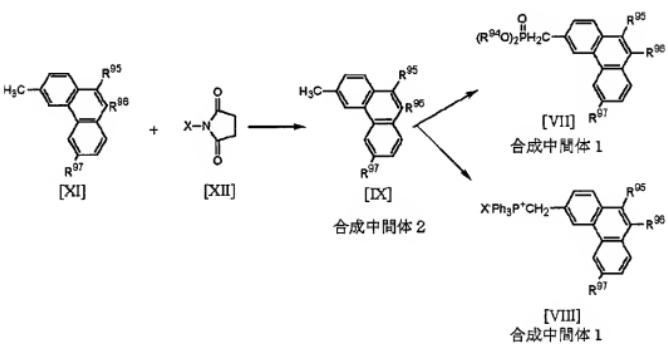
[Formula

87]

Reaction

scheme

2



[0033] Drawing 10 - drawing 13 show the example of the organic electroluminescence devices (EL element) which use the compound of this invention as an organic luminescent material, respectively.

[0034] Drawing 10 is the transparency mold organic electroluminescence

devices A to which the luminescence light 20 penetrates cathode 3, and the luminescence light 20 can be observed also from a protective layer 4 side.

Drawing 11 shows the reflective mold organic electroluminescence devices B which also obtain the reflected light in cathode 3 as a luminescence light 20.

[0035] Among drawing, one is a substrate for forming organic electroluminescence devices, and can use glass, plastics, and other proper ingredients. Moreover, when a substrate can also be shared when using organic electroluminescence devices combining other display devices, for example, carrying out an active-matrix drive, it is also possible to use TFT (Thin Film Transistors: thin film transistor) as a substrate. 2 is a transparent electrode (anode plate), can use transparent electrodes ITO (Indium tin oxide) and IZO (Indium zinc oxide) and SnO<sub>2</sub> grade in the above-mentioned transparency mold organic electroluminescence devices A, and can use Cr, Fe, Co, nickel, Cu, Ta, W, Pt, Mo, Au(s), these alloys, etc. in the reflective mold organic electroluminescence devices B.

[0036] Moreover, 5 is an organic luminous layer and contains the compound of this invention as a luminescent material. About this luminous layer, well-known various configurations can be conventionally used as lamination which obtains organic electroluminescence 20. When the ingredient which constitutes an electron hole transportation layer or an electron transport layer has a

luminescence so that it may mention later for example, the structure which carried out the laminating of these thin films can be used. Furthermore, in order to raise charge transportability ability in the range which fills the purpose of this invention, both an electron hole transportation layer, and both [ either or ] bar using the structure which carried out the laminating of the thin film of two or more sorts of ingredients, or the thin film which consists of a presentation which mixed two or more sorts of ingredients. Moreover, in order to improve the luminescence engine performance, the ingredient of at least one or more sorts of fluorescence may be used, and the structure which pinched this thin film between the electron hole transportation layer and the electron transport layer, and the structure where the ingredient of at least one or more sorts of fluorescence was further included in an electron hole transportation layer, electron transport layers, or these both may be used. In order to improve luminous efficiency in these cases, it is also possible to include the thin film for controlling transportation of an electron hole or an electron in the lamination.

[0037] Since the compound of this invention has both electronic transportability ability and electron hole transportability ability, it can be used during a component configuration also as a luminous layer which served as the electron hole transportation layer also as a luminous layer which served both as the electron transport layer. Moreover, it is also possible to consider as the

configuration put in the electron transport layer and the electron hole transportation layer by making the compound of this invention into a luminous layer.

[0038] In addition, among drawing 10 and drawing 11, three are cathode and can use the structure which carried out the laminating of the alloy of a metal [ activity / calcium / Li, Mg, ] and metals, such as Ag, aluminum, and In, LiF and LiO<sub>2</sub>, or these as an electrode material. In the organic electroluminescence devices of a transparency mold, the light transmittance suitable for an application can be obtained by adjusting the thickness of cathode. on the other hand -- the organic electroluminescence devices of a reflective mold -- setting -- the thickness of cathode -- thin -- carrying out -- high permeability -- holding -- in addition -- and organic electroluminescence can be taken out to a cathode side by constituting an anode plate from an ingredient with a high reflection factor. Moreover, four in drawing is the closure and a protective layer, and the effectiveness goes up it by making the organic whole electroluminescence devices into wrap structure. A proper ingredient can be used if airtightness is maintained. Moreover, 8 is a drive power source for current impregnation.

[0039] In the organic electroluminescence devices based on this invention, the organic layer has the organic laminated structure (it is terrorism structure to a single) to which the laminating of an electron hole transportation layer and the

electron transport layer was carried out, and the compound of this invention may be used as a formation ingredient of an electron hole transportation layer or an electron transport layer. Or the organic layer has the organic laminated structure (it is terrorism structure to double) to which the laminating of an electron hole transportation layer, a luminous layer, and the electron transport layer was carried out one by one, and the compound of this invention may be used as a formation ingredient of a luminous layer.

[0040] When the example of the organic electroluminescence devices which have such an organic laminated structure is shown, drawing 12 has the laminated structure to which the laminating of organic layer 5a which consists of an anode plate 2, and the electron hole transportation layer 6 and electron transport layer 7 of translucency on the penetrable substrate 1, and the cathode 3 was carried out one by one, and this laminated structure is the organic electroluminescence devices C of terrorism structure in the single a protective coat 4 comes for the closure to be carried out.

[0041] In the case of the lamination which omitted the luminous layer as shown in drawing 12 , the luminescence light 20 of predetermined wavelength is generated from the interface of the electron hole transportation layer 6 and an electron transport layer 7. Such luminescence light is observed from a substrate 1 side.

[0042] Moreover, drawing 13 has the laminated structure to which the laminating of organic layer 5b which consists of an anode plate 2, and the electron hole transportation layer 10, the luminous layer 11 and electron transport layer 12 of translucency on the substrate 1 of translucency, and the cathode 3 was carried out one by one, and this laminated structure is the organic electroluminescence devices D of terrorism structure in the double a protective coat 4 comes for the closure to be carried out.

[0043] In the organic electroluminescence devices shown in drawing 13 , the electron with which the electron hole poured in from the anode plate 2 was poured in from cathode 3 through the electron hole transportation layer 10 reaches a luminous layer 11 through an electron transport layer 12, respectively by impressing direct current voltage between an anode plate 2 and cathode 3. Consequently, the recombination of an electron/electron hole arises in a luminous layer 11, a singlet exciton generates, and luminescence of predetermined wavelength is generated from this singlet exciton.

[0044] In each organic electroluminescence devices C and D mentioned above, the ingredient of light transmission nature, such as glass and plastics, can be suitably used for a substrate 1. Moreover, when using combining other display devices, or when arranging the laminated structure shown in drawing 12 and drawing 13 in the shape of a matrix, it is good considering this substrate as

common use. Moreover, Components C and D can all take any structure of a transparency mold and a reflective mold.

[0045] Moreover, an anode plate 2 is a transparent electrode and ITO (indium tin oxide) and SnO<sub>2</sub> grade can be used for it. Between this anode plate 2 and the electron hole transportation layer 6 (or electron hole transportation layer 10), the thin film which consists of the organic substance or an organometallic compound may be prepared in order to improve the injection efficiency of a charge. In addition, when the protective coat 4 is formed with conductive ingredients, such as a metal, the insulator layer may be prepared in the side face of an anode plate 2.

[0046] Moreover, the electron hole transportation layer 6 and an electron transport layer 7 are organic layers by which the laminating was carried out, the compound of this invention contains organic layer 5a in the organic electroluminescence devices C to these either or both sides, and it is good as the luminescent electron hole transportation layer 6 or a luminescent electron transport layer 7. Organic layer 5b in the organic electroluminescence devices D can take various laminated structures, although the electron hole transportation layer 10, the luminous layer 11 containing the compound of this invention, and an electron transport layer 12 are organic layers by which the laminating was carried out. For example, both the electron hole transportation layer, and both

[ either or ] may have a luminescence.

[0047] Moreover, although it is desirable that it is the layer which the electron hole transportation layer 6 or an electron transport layer 7, and a luminous layer 11 turn into from the compound of this invention especially, these layers may be formed only with the compound of this invention, or you may form by the compound of this invention, other electron holes, or vapor codeposition with electronic transportation ingredients (for example, aromatic amine and pyrazolines etc.). Furthermore, in an electron hole transportation layer, in order to raise electron hole transportability ability, the electron hole transportation layer which carried out the laminating of two or more sorts of electron hole transportation ingredients may be formed.

[0048] Moreover, in the organic electroluminescence devices C, although a luminous layer may be the electronic transportability luminous layer 7, depending on the electrical potential difference impressed from a power source 8, light may be emitted by the electron hole transportation layer 6 or its interface. Similarly, in the organic electroluminescence devices D, a luminous layer may be an electron transport layer 12 in addition to layer 11, and may be the electron hole transportation layer 10. In order to raise the luminescence engine performance, it is good that it is the structure where the luminous layer 11 which used at least one sort of fluorescence ingredients was made to pinch between

an electron hole transportation layer and an electron transport layer. Or the structure where an electron hole transportation layer, an electron transport layer, or both [ these ] layers were made to contain this fluorescence ingredient may be constituted. In such a case, in order to improve luminous efficiency, it is also possible to include the thin films (a hole blocking layer, exciton generation layer, etc.) for controlling transportation of an electron hole or an electron in the lamination.

[0049] Moreover, you may be the structure in which could use the alloy of a metal [ activity / calcium / Li, Mg, ] and metals, such as Ag, aluminum, and In, as an ingredient used for cathode 3, and these metal layers carried out the laminating. In addition, the organic electroluminescence devices corresponding to an application are producible by choosing the thickness and the quality of the material of cathode suitably.

[0050] Moreover, a protective coat 4 acts as closure film, is making the organic whole electroluminescence devices into wrap structure, and can improve charge injection efficiency and luminous efficiency. In addition, if the airtightness is maintained, a single metal or alloys, such as aluminum, gold, and chromium, etc. can choose the ingredient suitably.

[0051] Although the current impressed to each above-mentioned organic electroluminescence devices is usually a direct current, pulse current and an

alternating current may be used. If a current value and an electrical-potential-difference value are within the limits which does not mind a component, there will be especially no limit, but when the power consumption and the life of organic electroluminescence devices are taken into consideration, it is desirable to make light emit efficiently with as small electrical energy as possible.

[0052] Next, drawing 14 is the example of a configuration of the flat-surface display which used the organic electroluminescence devices of this invention. In the full color display, like illustration, red (R) and the green organic layer 5 (5a, 5b) which can emit light in the three primary colors of (G) and blue (B) are allotted between cathode 3 and an anode plate 2. It can prepare in the shape of [ which crosses mutually ] a stripe, it is chosen by the luminance-signal circuit 14 and the control circuit 15 with a built-in shift register, and a signal level is impressed to each, and cathode 3 and an anode plate 2 are constituted so that the organic layer of the location (pixel) where the cathode 3 and the anode plate 2 which were chosen by this cross may emit light. As this drive approach, a simple matrix method or an active-matrix method can be used.

[0053] That is, it is a 8x3RGB simple matrix, and drawing 14 arranges the layered product 5 which consists of one side between cathode 3 and an anode plate 2, even if there are few electron hole transportation layers, and luminous

layers and electron transport layers either (refer to drawing 12 or drawing 13 ).

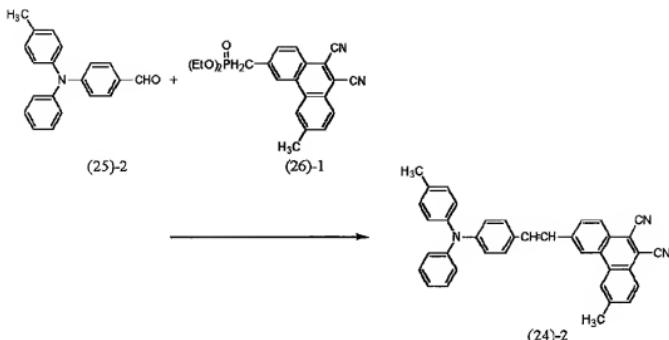
Both cathode and an anode plate are made to go direct in the shape of a matrix mutually, impress a signal level serially by the control circuits 15 and 14 with a built-in shift register, and they are constituted so that light may be emitted in the decussation location, while carrying out patterning to the shape of a stripe. Of course, the EL element of this configuration can be used also as picture reproducer as a display of an alphabetic character, a signal, etc. Moreover, the stripe-like pattern of cathode 3 and an anode plate 2 is arranged for every color of red (R), green (G), and blue (B), and it becomes possible to constitute multicolor or all full color solid-state mold flat-panel displays.

[0054]

[Example] Although this invention is concretely explained about an example below, this invention is not limited to the following examples.

[0055] Example 1 <example of composition of amino styryl phenanthrene compound (structure expression (24) -2)> [0056]

[Formula 88]



[0057] 0.194g (a mineral oil is entered) (4.86mmol) of sodium hydride is measured in a reaction container. Wash twice by the hexane and anhydrous tetrahydrofuran 10mL is made to suspend. On an ice bath, Under nitrogen-gas-atmosphere mind, a Wittig-Horner reagent () (26) -10.583g (4.62mmol) and a 4-[N-(methylphenyl)-N-phenyl] amino benzaldehyde ((25) -2) 0.511g (1.78mmol) anhydrous tetrahydrofuran : N.N-dimethylformamide =85:15 mixed-solution 100mL is dropped over 15 minutes. It agitated at 0 degree C then for 6 hours. Reaction mixed liquor was quenched on little ice, and after extracting with toluene and washing with saturation brine, it dried on anhydrous sodium sulfate.

[0058] The silica gel chromatography (Wako gel C-300, toluene:chloroform = 2:1) refined, from the toluene-hexane, it recrystallized 4 times and 0.572g of red crystals was obtained. [0059] The specified substance was identified by 1H

NMR and FAB-MS measurement. 67% of yield.

1H NMR (CDCl3) delta (ppm): 2.35 (s, 3H), 2.72 (s, 3H), and 7.03- 7.50 (m, 15H),

7.67 (d, 1H), 8.98 (d, 1H), 8.25 (m, 2H), 8.54 (s, 1H), and 8.63 (s, 1H) [0060]

Although the melting point was 280 degrees C as a result of the thermal analysis

by DSC (differential scanning calorimetry: differential scanning calorimetry), the

glass transition point was not observed. 453nm and the fluorescence maximum

wave length of the visible absorption maximum of a tetrahydrofuran solution

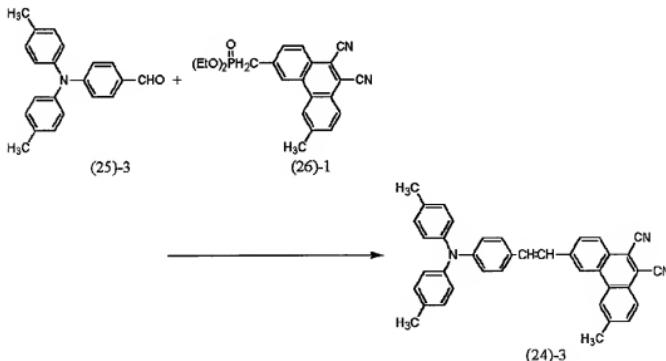
were 643nm. Moreover, the 1H NMR spectrum was as being shown in drawing

1.

[0061] Example 2 <example of composition of amino styryl phenanthrene

compound ((24)-3)> [0062]

[Formula 89]



[0063] 0.240g (a mineral oil is entered) (6.00mmol) of sodium hydride is measured in a reaction container. Wash twice by the hexane and anhydrous tetrahydrofuran 5mL is made to suspend. On an ice bath, Under nitrogen-gas-atmosphere mind, a Wittig-Horner reagent () (26) anhydrous tetrahydrofuran:N,N-dimethylformamide =85:15 mixed-solution 100mL (-10.721g (2.00mmol) and 4-[N and N-JI (methylphenyl)] amino benzaldehyde ((25) -3) 0.661g (2.20mmol)) It was dropped over 15 minutes and agitated at 0 degree C as it is for 6 hours. Reaction mixed liquor was quenched on little ice, and after extracting with toluene and washing with saturation brine, it dried on anhydrous sodium sulfate.

[0064] The silica gel chromatography (Wako gel C-300, toluene:chloroform = 1:1) refined, from the toluene-hexane, it recrystallized 4 times and 0.569g of red crystals was obtained. [0065] The specified substance was identified by <sup>1</sup>H NMR and FAB-MS measurement. 53% of yield.

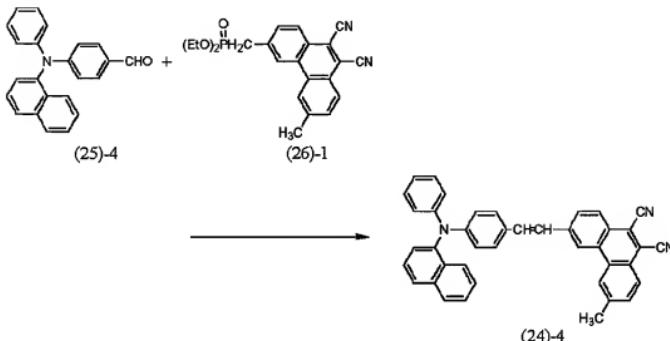
<sup>1</sup>H NMR (CDCl<sub>3</sub>) delta (ppm):2.34 (s, 6H), 2.72 (s, 3H), and 7.01- 7.39 (m, 12H), 7.44 (d, 2H), 7.67 (d, 1H), 8.01 (d, 1H), 8.28 (m, 2H), 8.55 (s, 1H), and 8.65 (s, 1H) [0066] Although the melting point was 312 degrees C as a result of the thermal analysis by DSC, the glass transition point was not observed. 460nm

and the fluorescence maximum wave length of the visible absorption maximum of a tetrahydrofuran solution were 667nm. Moreover, the <sup>1</sup>H NMR spectrum was

as being shown in drawing 2 .

[0067] Example 3 <example of composition of amino styryl phenanthrene compound ((24)-4)> [0068]

[Formula 90]



[0069] 0.308g (a mineral oil is entered) (7.77mmol) of sodium hydride is measured in a reaction container. Wash twice by the hexane and anhydrous tetrahydrofuran 20mL is made to suspend. On an ice bath, Under nitrogen-gas-atmosphere mind Wittig-Horner () (26) 0.925g of -1 reagents (2.57mmol) and a 4-[N-(1-naphthyl)-N-phenyl] amino benzaldehyde ((25)-4) 1.16g (3.59mmol) anhydrous tetrahydrofuran : N.N-dimethylformamide =9:1 mixed-solution 100mL is dropped over 15 minutes. It stirred at the room temperature further by 0 degree C then for 12 hours for 6 hours. Reaction mixed liquor was quenched on little ice, and after extracting with toluene and washing

with saturation brine, it dried on anhydrous sodium sulfate.

[0070] After alumina chromatography (200 meshes and a toluene:tetrahydrofuran = 1:1) refined, flash chromatography (Merk Silica Gel HF254, toluene) refined, it recrystallized [ hexane / toluene-] 3 times, and red crystal (7)0.310g was obtained.

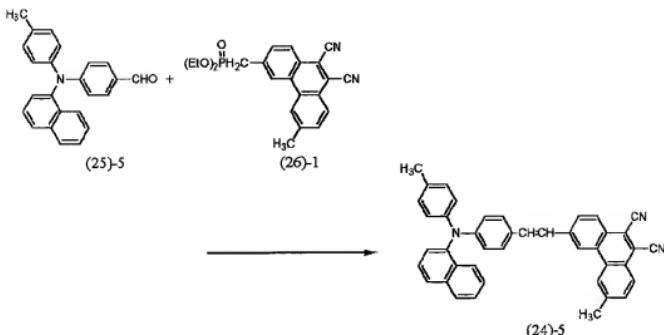
[0071] The specified substance was identified by 1H NMR and FAB-MS measurement. 17% of yield.

1H NMR (CDCl<sub>3</sub>) delta (ppm):2.72 (s, 3H), 6.97-7.54 (m, 15H), 7.67 (d, 1H), 7.82 (d, 1H), 7.97 (m, 3H), 8.27 (m, 2H), 8.54 (s, 1H), 8.63 (s, 1H) [0072]

Although the melting point was 315 degrees C as a result of the thermal analysis by DSC, the glass transition point was not observed. 450nm and the fluorescence maximum wave length of the visible absorption maximum of a tetrahydrofuran solution were 631nm. Moreover, the 1H NMR spectrum was as being shown in drawing 3 .

[0073] Example 4 <example of composition of amino styryl phenanthrene compound ((24) -5)> [0074]

[Formula 91]



[0075] 0.194g (a mineral oil is entered) (4.86mmol) of sodium hydride is measured in a reaction container. Wash twice by the hexane and anhydrous tetrahydrofuran 10mL is made to suspend. On an ice bath, Under nitrogen-gas-atmosphere mind Wittig-Horner () (26) 0.583g of -1 reagents (1.62mmol) and a 4-[N-(methylphenyl)-N-(1-naphthyl)] amino benzaldehyde ((25) -5) 0.600g (1.78mmol) anhydrous tetrahydrofuran : N.N-dimethylformamide =85:15 mixed-solution 100mL It was dropped over 15 minutes and stirred at 0 degree C as it is for 6 hours. Reaction mixed liquor was quenched on little ice, and after extracting with toluene and washing with saturation brine, it dried on anhydrous sodium sulfate.

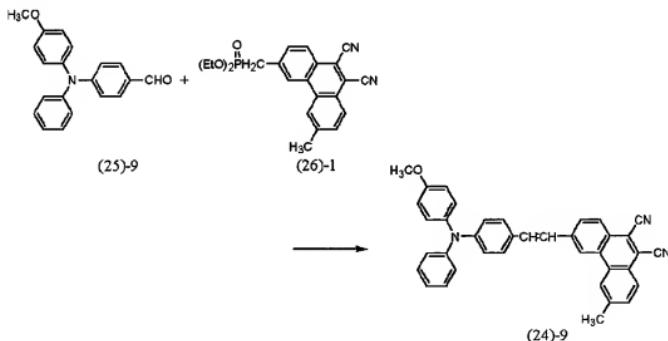
[0076] The silica gel chromatography (Wako gel C-300, toluene:chloroform = 2:1) refined, from the toluene-hexane, it recrystallized 4 times and 0.657g of red crystals was obtained. [0077] The specified substance was identified by 1H

NMR and FAB-MS measurement. 70% of yield.

1H NMR (CDCl3) delta (ppm):2.33 (s, 3H), 2.72 (s, 3H), and 6.92- 7.53 (m, 14H), 7.67 (d, 1H), 7.81 (d, 1H), and 7.90- 8.02 (m, 3H), 8.28 (m, 2H), 8.55 (s, 1H), and 8.65 (s, 1H) [0078] Although the melting point was 320 degrees C as a result of the thermal analysis by DSC, the glass transition point was not observed. 455nm and the fluorescence maximum wave length of the visible absorption maximum of a tetrahydrofuran solution were 634nm. Moreover, the 1H NMR spectrum was as being shown in drawing 4 .

[0079] Example 5 <example of composition of amino styryl phenanthrene compound ((24)-9)> [0080]

[Formula 92]



[0081] 0.183g (a mineral oil is entered) (4.58mmol) of sodium hydride is measured in a reaction container. Wash twice by the hexane and anhydrous

tetrahydrofuran 20mL is made to suspend. On an ice bath, Under nitrogen-gas-atmosphere mind, a Wittig-Horner reagent () (26) -10.505g (2.57mmol) and a 4-[N-(methoxyphenyl)-N-phenyl] amino benzaldehyde ((25) -9) 0.511g (1.68mmol) anhydrous tetrahydrofuran : N,N-dimethylformamide =85:15 mixed-solution 100mL It was dropped over 15 minutes and agitated at 0 degree C as it is for 6 hours. Reaction mixed liquor was quenched on little ice, and after extracting with toluene and washing with saturation brine, it dried on anhydrous sodium sulfate.

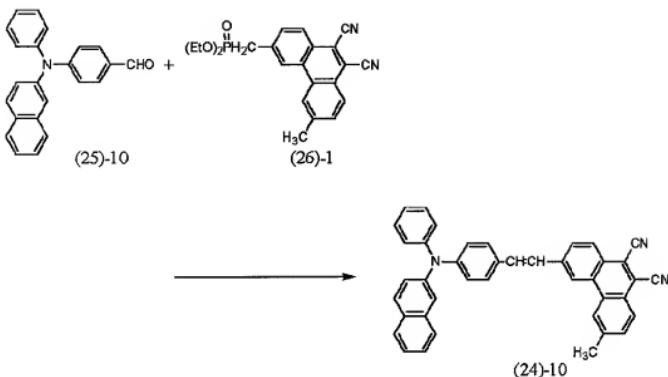
[0082] The silica gel chromatography (Wako gel C-300, toluene) refined, from the toluene-hexane, it recrystallized 4 times and 0.574g of red crystals was obtained. [0083] The specified substance was identified by 1H NMR and FAB-MS measurement. 69% of yield.

1H NMR (CDCl<sub>3</sub>) delta (ppm):2.72 (s, 3H), 3.83 (s, 3H), 6.88 (d, 2H), 6.90-7.40 (m, 11H), 7.46 (d, 2H), 7.67 (d, 1H), 8.01 (d, 1H), 8.28 (m, 2H), 8.55 (s, 1H), 8.65 (s, 1H) [0084] Although the melting point was 277 degrees C as a result of the thermal analysis by DSC, the glass transition point was not observed. 459nm and the fluorescence maximum wave length of the visible absorption maximum of a tetrahydrofuran solution were about 690nm. Moreover, the 1H NMR spectrum was as being shown in drawing 5 .

[0085] Example 6 <example of composition of amino styryl phenanthrene

compound ((24)-10)> [0086]

[Formula 93]



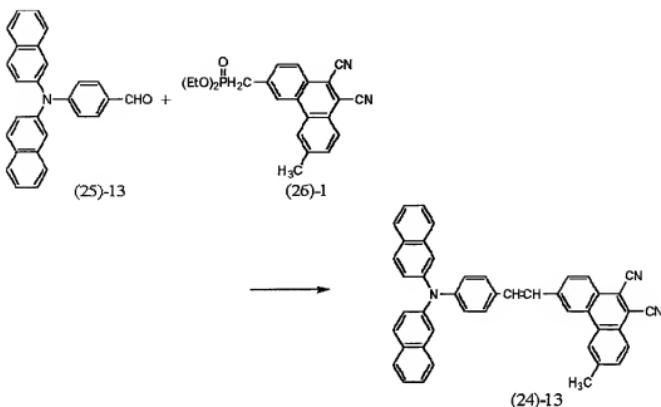
[0087] 0.167g (a mineral oil is entered) (4.17mmol) of sodium hydride is measured in a reaction container. Wash twice by the hexane and anhydrous tetrahydrofuran:anhydrous dimethylformamide =85:15 mixed-solution 10mL is made to suspend. On an ice bath, Under nitrogen-gas-atmosphere mind, a Wittig-Horner reagent () (26) -10.500g (1.39mmol) and a 4-[N-phenyl-N-(2-naphthyl)] amino benzaldehyde ((25)-10) 0.494g (1.53mmol) anhydrous tetrahydrofuran : N,N-dimethylformamide =85:15 mixed-solution 90mL is dropped over 15 minutes. It stirred at 0 degree C then for 3 hours. Reaction mixed liquor was quenched on little ice, and after extracting with toluene and washing with saturation brine, it dried on anhydrous sodium sulfate.

[0088] The silica gel chromatography (Wako gel C-300, toluene:chloroform = 2:1) refined, from the toluene-hexane, it recrystallized 4 times and 0.531g of red crystals was obtained. [0089] The specified substance was identified by 1H NMR and FAB-MS measurement. 68% of yield.

1H NMR (CDCl<sub>3</sub>) delta (ppm) : 2.72 (s, 3H), 7.11-7.44 (m, 12H), 7.52 (m, 3H), 7.67 (m, 2H), 7.79 (m, 2H), 8.02 (d, 1H), 8.25 (d, 1H), 8.30 (d, 1H), 8.56 (s, 1H), 8.66 (s, 1H) [0090] As a result of the thermal analysis by DSC, the melting point was 284 degrees C and the glass transition point was 154 degrees C. 454nm and the fluorescence maximum wave length of the visible absorption maximum of a tetrahydrofuran solution were 647nm. Moreover, the 1H NMR spectrum was as being shown in drawing 6 .

[0091] Example 7 <example of composition of amino styryl phenanthrene compound ((24) -13)> [0092]

[Formula 94]



[0093] 0.167g (a mineral oil is entered) (4.17mmol) of sodium hydride is measured in a reaction container. Wash twice by the hexane and anhydrous tetrahydrofuran:anhydrous dimethylformamide =85:15 mixed-solution 10mL is made to suspend. On an ice bath, Under nitrogen-gas-atmosphere mind, a Wittig-Horner reagent An anhydrous tetrahydrofuran (0.500g (1.39mmol) and 4-screw [N-(2-naphthyl)] amino benzaldehyde ((25)-13) 0.571g (1.53mmol)) : ((26)-1) N,N-dimethylformamide =85:15 mixed-solution 90mL It was dropped over 15 minutes and stirred at 0 degree C as it is for 2 hours. Reaction mixed liquor was quenched on little ice, and after extracting with toluene and washing with saturation brine, it dried on anhydrous sodium sulfate.

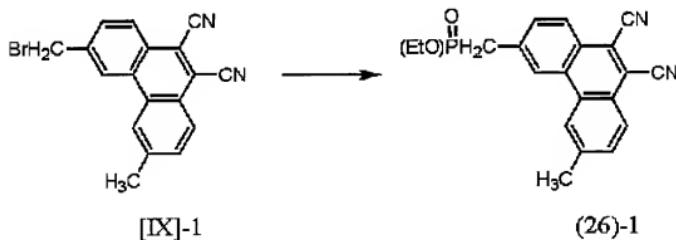
[0094] The silica gel chromatography (Wako gel C-300, toluene:chloroform =

2:1) refined, from the toluene-hexane, it recrystallized 4 times and 0.635g of red crystals was obtained. [0095] The specified substance was identified by 1H NMR and FAB-MS measurement. 75% of yield.

1H NMR ( $\text{CDCl}_3$ ) delta (ppm): 2.71 (s, 3H), 7.19 (d, 2H), 7.31-7.45 (m, 7H), 7.51-7.82 (m, 12H), 8.02 (d, 1H), 8.27 (d, 1H), 8.32 (d, 1H), 8.55 (s, 1H), 8.68 (s, 1H) [0096] As a result of the thermal analysis by DSC, the melting point was 317 degrees C and the glass transition point was 193 degrees C. 455nm and the fluorescence maximum wave length of the visible absorption maximum of a tetrahydrofuran solution were 639nm. Moreover, the 1H NMR spectrum was as being shown in drawing 7.

[0097] Example 8 <example of composition of phosphonate ((26) -1)> [0098]

[Formula 95]



-1) 0.860g (2.57mmol) and phosphorous acid triethyl 3.28g (19.8mmol) were

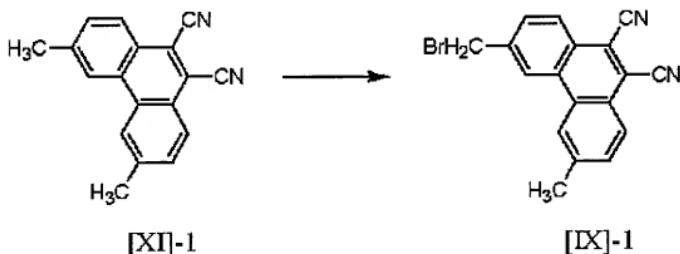
made to suspend in xylene 150mL, it flowed back quietly for 24 hours, and the specified substance was obtained quantitatively.

[0100] The specified substance was identified by  $^1\text{H}$  NMR and FAB-MS measurement.

$^1\text{H}$  NMR ( $\text{CDCl}_3$ ) delta (ppm):1.31 (t, 6H), 2.70 (s, 3H), 3.47 (d, 2H), 4.07 (q, 4H), 7.63 (m, 1H), 7.78 (m, 1H), 8.28 (m, 2H), 8.51 (d, 1H), and a 8.70(brs, 1H)  $^1\text{H}$  NMR spectrum were as being shown in drawing 8 .

[0101] Example 9 <example of composition of 3-(bromomethyl)-6-methyl phenanthrene 9 and 10-JIKARUBO nitril ([IX]-1)> [0102]

[Formula 96]

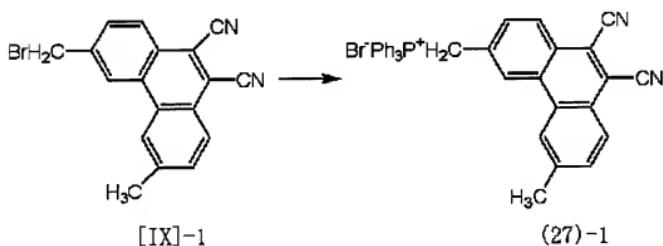


[0103] After dissolving 3, 6-dimethyl phenanthrene -9, and 10-JIKARUBO nitril ([XI]-1) 0.800g (3.12mmol) in chloroform 200mL and performing oxygen degassing through nitrogen, 0.556g (3.12mmol) of N-bromosuccinimide was added, and it flowed back for 6 hours. the precipitate obtained by adding

NBS0.556g (3.12mmol) furthermore, flowing back for 6 hours, condensing a reaction solution to 50mL(s) and carrying out filtration under reduced pressure -- ethanol -- it often washed by the hexane continuously and 0.860g of specified substance was obtained. [0104] The specified substance was identified by 1H NMR and FAB-MS measurement. 82% of yield.

1H NMR (CDCl<sub>3</sub>) delta (ppm):4.77 (S, 2H), 7.68 (m, 1H), 7.90 (m, 1H), 8.30 (m, 1H), 8.37 (m, 1H), 8.53 (s, 1H), and a 8.73(s, 1H) 1H NMR spectrum were as being shown in drawing 9.

[0105] Example 10 <example of composition of phosphonium ((27) -1)> [0106]

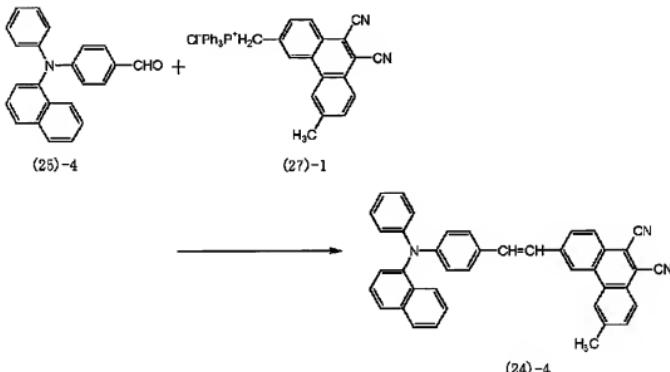


[0107] 3-(bromomethyl)-6-methyl phenanthrene 9 and 10-JIKARUBO nitril ([IX])  
-1) 0.860g (2.57mmol) and triphenyl phosphine 3.37g (12.9mmol) -- xylene  
150ml -- it was made to suspend in inside and flowed back quietly for 24 hours.  
The reaction solution was condensed in one half, the produced precipitate was

filtered, and the specified substance was obtained quantitatively. 1H The specified substance was identified by NMR and FAB-MS measurement.

[0108] Example 11 <example of composition of amino styryl phenanthrene compound ((24)-4)> [0109]

[Formula 98]



[0110] 0.167g (a mineral oil is entered) (4.17mmol) of sodium hydroxides is measured in a reaction container. Wash twice by the hexane and 10ml of anhydrous tetrahydrofuran:anhydrous dimethylformamide =85:15 mixed solutions is made to suspend. On an ice bath, Under nitrogen-gas-atmosphere mind, a Witting reagent () 0.830g (27-1) (1.39mmol) and a 4-[N-phenyl-N-(1-naphthyl)] amino benzaldehyde ((25)-4) 0.494g (1.53mmol) anhydrous tetrahydrofuran : 90ml of N,N-dimethylformamide =85:15 mixed

solutions is dropped over 15 minutes. It stirred at 0 degree C then for 3 hours.

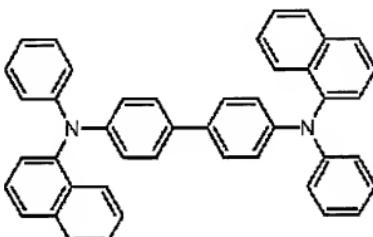
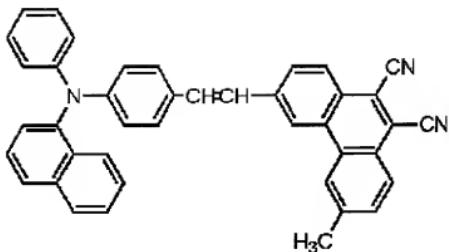
Reaction mixed liquor was quenched on little ice, and after extracting with toluene and washing with saturation brine, it dried on anhydrous sodium sulfate.

[0111] The silica gel chromatography (Wako gel C-300, toluene:chloroform = 2:1) refined, from the toluene-hexane, it recrystallized 4 times and 0.318g of red crystals was obtained. 1H By NMR and FAB-MS measurement, the same spectrum as (24)-4 (example 3) compounded by the Wittig-Horner reaction was obtained, and the specified substance was identified (41% of yield).

[0112] Example 12 this example is an example which produced single hetero structure transparency mold organic electroluminescence devices for the compound of following structure-expression (24)-4, using an electronic transportability luminous layer and 4, and 4'-screw [N, N'-Jl (1-naphthyl)-N, and N'-diphenyl] biphenyl diamine (alpha-NPD) as an electron hole transportation layer.

[0113]

[Formula 99] Structure expression (24) -4 :



Alpha-NPD :

[0114] First, the 30mmx30mm glass substrate with which the anode plate which consists of ITO with a thickness of 100nm was formed on one front face into the vacuum evaporation system was set. The substrate was approached, the metal mask which has two or more 2.0mmx2.0mm unit openings as a vacuum evaporationo mask has been arranged, and alpha-NPD which is an electron hole transportation ingredient under the vacuum of 10<sup>-4</sup> or less Pa was vapor-deposited in thickness of 50nm with the vacuum deposition method. Furthermore, the above-mentioned structure-expression (24)-4 [ 50nm ] were vapor-deposited in contact with the electron hole transportation layer as an

electronic transportability luminescent material. The vacuum evaporationo rate was made respectively in 0.2nm/second.

[0115] The cascade screen of Mg and Ag was adopted as a cathode material, and by vacuum evaporationo, it formed in the thickness of 50nm (Mg film) and 150nm (Ag film) as vacuum evaporationo rate 1nm/a second, and although this also showed drawing 12 by the example 10, it produced \*\*\*\* organic electroluminescence devices.

[0116] Thus, forward bias direct current voltage was applied to the organic electroluminescence devices of the produced example 10 under nitrogen-gas-atmosphere mind, and the luminescence property was evaluated. The luminescent color was red, and as a result of performing spectrometry, it obtained the spectrum which has a luminescence peak near 637nm. The spectroscope which used the photodiode array by the Otsuka electronic company as the detector was used for spectrometry. Moreover, when the electrical-potential-difference-measurement of luminance was performed, the brightness of 1000 cd/m<sup>2</sup> was obtained by 8V.

[0117] After producing these organic electroluminescence devices, although it was left for one month under nitrogen-gas-atmosphere mind, component degradation was not observed. Moreover, when energizing a current value uniformly, carrying out continuation luminescence and carrying out forcible

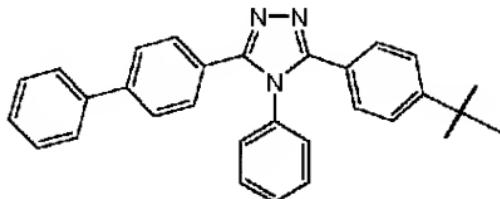
degradation by initial brightness 200 cd/m<sup>2</sup>, it was 900 hours until brightness was halved.

[0118] Example 13 this example said compound of structure-expression (24)-4 A luminous layer, 4 expressed with said structure expression, and 4'-screw [N, N-JI (1-naphthyl)-N, and N'-diphenyl] biphenyl diamine (alpha-NPD) An electron hole transportation layer, And it is the example which created terrorism structure transparency mold organic electroluminescence devices to double, using the 2-(4-biphenyl)-5-(4-tert-butylphenyl)-1-phenyls 1 and 3 and 4-triazole (TAZ) which are expressed with the following structure expression as an electron transport layer ingredient.

[0119]

[Formula 100]

TAZ :



[0120] First, the 30mmx30mm glass substrate with which the anode plate which consists of ITO with a thickness of 100nm was formed on one front face into the vacuum evaporation system was set. The substrate was approached, the metal

mask which has two or more 2.0mmx2.0mm unit openings as a vacuum evaporationo mask has been arranged, and alpha-NPD which is an electron hole transportation ingredient under the vacuum of 10 - 4 or less Pa was vapor-deposited in thickness of 50nm with the vacuum deposition method. Next, the compound expressed with said structure-expression (24)-4 as a luminescent material was vapor-deposited in thickness of 25nm in contact with the electron hole transportation layer. Furthermore, TAZ as an electronic transportation ingredient was vapor-deposited in thickness of 25nm in contact with the luminous layer. The vacuum evaporationo rate was made respectively in 0.2nm/second.

[0121] The cascade screen of Mg and Ag was adopted as a cathode material, and by vacuum evaporationo, this was also formed in the thickness of 50nm (Mg film) and 150nm (Ag film) as vacuum evaporationo rate 1nm/a second, and produced organic electroluminescence devices as shown in drawing 13 by the example 13.

[0122] Thus, forward bias direct current voltage was applied to the organic electroluminescence devices of the produced example 13 under nitrogen-gas-atmosphere mind, and the luminescence property was evaluated. The luminescent color was red, and as a result of performing spectrometry, it obtained the spectrum which has a luminescence peak near 637nm. The

spectroscope which used the photodiode array by the Otsuka electronic company as the detector was used for spectrometry. Moreover, when the electrical-potential-difference-measurement of luminance was performed, the brightness of 2000 cd/m<sup>2</sup> was obtained by 8V.

[0123] After creating these organic electroluminescence devices, although it was left for one month under nitrogen-gas-atmosphere mind, component degradation was not observed. Moreover, when energizing a current value uniformly, carrying out continuation luminescence and carrying out forcible degradation by initial brightness 200 cd/m<sup>2</sup>, it was 1100 hours until brightness was halved.

[0124]

[Function and Effect of the Invention] The amino styryl phenanthrene compound of this invention can be effectively used depending on the substituent introduced into the structure as an organic luminescent material which shows strong luminescence of the red from which luminescence maximum wave length differs, it is the matter which has the high melting point, and it excels in electric, thermal, or chemical stability, and it is amorphous and a vitreous state can be formed easily, and also has sublimability and can also form the uniform amorphous film with vacuum deposition etc. while excelling in thermal resistance. Moreover, the compound of this invention can be manufactured by the general and efficient approach through the synthetic intermediate field of this invention.

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## DESCRIPTION OF DRAWINGS

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[Brief Description of the Drawings]

[Drawing 1] 1H of the compound by the example 1 of this invention It is an NMR spectrum Fig.

[Drawing 2] 1H of the compound which this invention twists example 2 It is an NMR spectrum Fig.

[Drawing 3] 1H of the compound by the example 3 of this invention It is an NMR spectrum Fig.

[Drawing 4] 1H of the compound by the example 4 of this invention It is an NMR spectrum Fig.

[Drawing 5] 1H of the compound by the example 5 of this invention It is an NMR spectrum Fig.

[Drawing 6] 1H of the compound by the example 6 of this invention It is an NMR spectrum Fig.

[Drawing 7] 1H of the compound by the example 7 of this invention It is an NMR spectrum Fig.

[Drawing 8] 1H of the compound by the example 8 of this invention It is an NMR

spectrum Fig.

[Drawing 9] 1H of the compound by the example 9 of this invention It is an NMR spectrum Fig.

[Drawing 10] It is the important section outline sectional view of the organic electroluminescence devices based on this invention.

[Drawing 11] It is the important section outline sectional view of organic electroluminescence devices besides \*\*\*\*.

[Drawing 12] It is the important section outline sectional view of organic electroluminescence devices besides \*\*\*\*.

[Drawing 13] It is the important section outline sectional view of \*\*\*\* and also other organic electroluminescence devices.

[Drawing 14] It is the block diagram of the full color flat-surface display using \*\*\*\* organic electroluminescence devices.

[Description of Notations]

1 [ ... A protective coat, 5, 5a, 5b / ... An organic layer, 6 / ... An electron hole transportation layer, 7 / ... An electron transport layer, 8 / ... A power source, 10 / ... An electron hole transportation layer, 11 / ... A luminous layer, 12 / ... An electron transport layer, 14 / ... A luminance-signal circuit, 15 / ... A control circuit, 20 / ... Luminescence light, A B C, D / ... Organic electroluminescence devices ] ... A substrate, 2 ... A transparent electrode (anode plate), 3 ... Cathode,

